The Boeing Company
P.O. Box 516
St. Louis, MO 63166-0516
(314) 232-0232 TELEX 44-857

107E-5974-JWH March 9, 2005

Ms. Demetra Salisbury U.S. Environmental Protection Agency Region VII 901 North Fifth Street Kansas City, KS 66101



Encl: TPH Soil Vapor Sampling Work Plan, Boeing Tract 1,

Hazelwood, Missouri (2 copies)

Dear Ms. Salisbury:

The enclosed soil vapor sampling work plan is submitted as required by Section XI of the Corrective Action Conditions of the Hazardous Waste Management Facility Permit, Number MOD 000 818 963. Three copies of the report have been submitted to the Missouri Department of Natural Resources as required under the terms of the permit.

Please contact me if you need additional information.

Sincerely,

Joseph W. Haake, Group Manager

Environmental and Hazardous Materials Services

Dept. 107E, Bldg. 111, Mailcode S111-2491

(314) 777-9181

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# TPH Soil Vapor Sampling Work Plan Boeing Tract 1 Hazelwood, Missouri

Prepared for: The Boeing Company St. Louis, Missouri



Prepared by:
MACTEC Engineering and Consulting, Inc.
3199 Riverport Tech Center Drive
St. Louis, Missouri 63043

MACTEC Project Number 3250035046.10

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Air Toxics Ltd

Missouri

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## 1.0 Purpose and Objective

A Resource Conservation and Recovery Act (RCRA) Facility Investigation [(RFI) MACTEC, 2004] has recently been completed at the Boeing Tract 1 facility (Facility) located in Hazelwood, Missouri (Figure 1-1). Soil borings, temporary piezometers and groundwater monitoring wells were installed at the Facility as part of the RFI to characterize the nature of any hazardous waste/constituent releases to soil or groundwater. Soil and groundwater samples were collected and selectively analyzed for volatile organic compounds (VOCs), total petroleum hydrocarbons (TPH), semi-volatile organic compounds (SVOCs), polychlorinated byphenols (PCBs) and total metals.

Following completion of the RFI, an assessment was conducted by Risk Assessment & Management Group Inc. (RAM Group) utilizing the RFI and other historical data and the Missouri Risk-Based Corrective Action (MRBCA) process. Based on this assessment, there was the potential for exceedence of acceptable risk at selected locations at the Facility (*Risk-Based Corrective Action Report, Boeing Tract 1*, September 2004).

For risk assessment purposes the site was divided into exposure units (nine Areas and 18 Sub-areas) based on the current and future land use and activity patterns (Figure 1-1). The intent of the exposure units was to define portions of the Facility that are relatively homogeneous in terms of risk and exposure factors and to develop target levels for each exposure unit consistent with the land use and/or other characteristics to the exposure condition of the Area.

At nine exposure units the risk assessment indicated that there was a potential for exposure to total petroleum hydrocarbons (TPH) diesel range organics (DRO); and/or oil range organics (ORO) by indoor inhalation from groundwater by a non-residential worker. Sub-area (3C) additionally exceeded the risk, for total TPH by outdoor inhalation from groundwater by a construction worker. Because these exceedences are primarily attributed to the very conservative models used to estimate the indoor inhalation vapor intrusion risk, soil vapor sampling was recommended as a better estimator of indoor vapor intrusion risk.

One of the nine exposure units that exceeded the risk for TPH is impacted as the result of off-site contamination; interim measures consisting of soil removal are planned at four others. Soil vapor sampling is proposed at the remaining four units (Risk Sub-areas 2B, 3C, 3G, and 6C).

Soil vapor sampling is needed to determine actual constituents of concern (COCs) concentrations to better estimate potential vapor intrusion into buildings and associated health risk to the building occupants. The results of this evaluation may indicate the absence of unacceptable health risk due to vapor intrusion.

Vapor samples will be analyzed for TPH and benzene, toluene, ethylbenzene, xylenes (BTEX), methyl tertiary-butyl ether (MTBE), and naphthalene. Additionally the vapor samples will be analyzed for the volatile COCs identified in the Risk Assessment for each Sub-area. Table 1-1 provides a listing of the COCs for each Sub-area.

## 2.0 Soil Vapor Sampling

## 2.1 Locations

TPH soil vapor sampling locations were determined by selecting the RFI groundwater sampling locations that contained the highest TPH concentrations for each Sub-area where a risk exceedence was calculated. A total of 14 sampling points were selected for the collection of soil vapor samples. These sampling points represent the highest TPH concentrations for each Risk Sub-area. Summaries of the boring locations selected are presented below and are marked on Figures 2-1 through 2-4.

### Risk Area 2B:

- TP-7
- TP-9
- TP-15
- TP-16

## Risk Area 3C:

- B42W1
- B42S5
- B45S8<sup>-</sup>
- B45S11

## Risk Area 3G:

Two locations in the vicinity of B2S2

#### Risk Area 6C:

- Two locations in the vicinity of B27E2
- Two locations in the vicinity of B27I9

## 2.2 Depth

Soil vapor samples will be collected approximately one foot above the capillary fringe, with a minimum collection depth of three feet below ground surface. Groundwater depth will be determined by inspection

of soil cores by the field geologist assisted by the measurement of groundwater at the nearest monitoring well to the sampling location. Historical groundwater depths for the nearest wells to the proposed sampling locations are presented in Table 2-1.

## 2.3 Frequency

The historical groundwater elevation data (Table 2-1) indicates that over a two year period the groundwater fluctuated 3 feet or less. Based on the constancy of the groundwater levels, soil vapor sampling will be a one time sampling event. The sampling event is proposed to occur in the late winter of 2005.

## 3.0 Soil Gas Measurement Techniques

Soil vapor sampling will follow the Standard Operating Procedures (SOP) for Soil Vapor Grab Sampling Activities Using the Geoprobe® Post-Run Tubing (PRT) System With 6-Liter SUMMA® Canisters and National Institute for Occupational Safety and Health (NIOSH) Tubes (RAM, 2004a). Soil vapor samples are collected in 6-Liter SUMMA® canisters and NIOSH tubes utilizing Geoprobe® direct-push boring methodologies and the PRT system. The target soil vapor sampling depth will be determined prior to sampling at each location as determined in Section 2.3 of the SOP. Optional field measurements for biogenic gases, such as oxygen, carbon dioxide, and methane, may be collected from each sampling location using a handheld landfill gas meter.

#### 3.1 Preparation

Prior to mobilization, MACTEC personnel will make all necessary preparations for the work. The preparations will include:

- Determine equipment accessibility to each sampling location,
- Coordinate schedule with subcontractors, property owner(s), regulatory agencies, and the laboratory,
- · Obtain all necessary field equipment and materials, and
- Verify that all necessary field equipment and materials were received and are properly operating.
   Refer to Appendix A (Guide to Air Sampling and Analysis, Air Toxics Ltd.) Section 2.3.2 before mobilization.

## 3.2 Direct-Push Geoprobe® Boring and Post-Run Tubing (PRT) Soil Vapor Sampling

This section describes the field methodology that will be utilized to collect the soil vapor samples. Because of site-specific conditions or obstacles, the sampling plan may be modified as needed, based on the professional judgment of the MACTEC field personnel.

MACTEC will utilize a Geoprobe® contractor to provide and operate the Geoprobe® rig and Geoprobe® equipment. The equipment will be positioned at the sampling location and the drive rod and expendable point will be advanced into the subsurface. Once the target sampling depth has been achieved, the rod is pulled up about 4 to 6 inches to allow the expendable point to be released and void space is created. It may be necessary to use a "point popper" to disengage the expendable point. Then the sampling tube with adapter is pushed down the inside of the drive rod and is engaged into the point holder by turning counter-clockwise. Once the tubing is engaged, the soil vapor sample can be collected using the PRT system. Detailed information for the PRT system is provided in Appendix B (Geoprobe® Post-Run Tubing System). Disposable Teflon® tubing (0.125-inch inside diameter) will be used for the collection of the soil vapor samples. Prior to collecting the sample, the sampling system (PRT tubing) will be purged of seven volumes of vapor. All calculations of required purge volumes will be documented in the field book. The sample train purge volume (one volume) will be calculated by the summation of the following volumes (refer to Appendix C for purge volume calculations):

- 1. The volume of Teflon® tubing utilized to extend from the target sampling depth to the surface,
- 2. The approximate volume of the void space created when the expendable point is withdrawn,
- 3. Plus an additional 20 milliliters (mL).

Purging of the sampling system will be performed using a 60 cubic centimeters (cc) [1 cc is equivalent to 1 mL] disposable syringe. A new syringe and new Teflon® tubing will be used for each sample collected.

SUMMA® canisters and NIOSH tubes will be used to collect the samples for laboratory analysis. Sections 2.3.1 and 2.3.2 in Appendix A provide a detailed description of the procedures to be used for SUMMA® canister grab sampling.

To confirm that ambient air short-circuiting is not occurring during soil vapor sampling, a leak test will be performed. The leak test includes using 2-propanol as a tracer compound. Household paper towels will be wetted with 2-propanol immediately prior to purging and sampling. The wetted paper towels are placed directly over the top of a bentonite plug located at ground surface where the Geoprobe® rod string enters the ground. A second wetted paper towel is placed around the Teflon® tubing where the tubing exits the rod string. When this short-circuiting test is conducted, 2-propanol will be added to the list of analytes to be analyzed by the laboratory. The procedures will be documented in the field logbook. Care will be exercised in the handling of 2-propanol to avoid cross-contaminating the samples or equipment. Marking pens (Sharpies®, etc) that may contain VOCs will not be used in the field.

Visual inspection of individual PRT system components, including O-rings, will be performed before and after collection of each soil vapor sample. Documentation of inspection is included in the field logbook. Qualitative testing of the equipment will be performed at the end of each day after the last sample is collected. The test is conducted by applying a vacuum to the sealed assembled system as described in Appendix D (Vacuum Testing Procedure).

## 3.2.1 Vapor Sample Collection using SUMMA® Canisters

The SUMMA® canisters sampling will be used to determine the concentrations of BTEX, MTBE, naphthalene, TPHg (calibrated), and 2-propanol (leak testing). The following steps will be followed for vapor sampling using the SUMMA® canisters:

- Prior to sampling, the system will be purged of seven volumes of ambient air using a GilAir Constant flow pump,
- Upon completion of purging the seventh purge volume, the in-line valve to the GilAir Constant flow pump will be closed,
- Insert the Teflon® tubing into a 5-micron particulate filter attached to a 6-Liter SUMMA® canister inlet,
- Then, to collect each sample, the field crew will perform the following steps:
  - First, open the valve on the SUMMA® canister, with the pressure gauge attached and record the canister pressure (should be approximately minus (-) 29 inches mercury),
  - > The grab sampling period will continue for approximately 3 minutes,
  - > Close the sample valve, and record the final canister pressure,
  - > If the final pressure is near ambient then remove the SUMMA® canister from pressure gauge and sample tubing,

- ➤ If the final pressure has not reached ambient, then reopen the canister and continue to collect soil gas. This procedure may need to be repeated until an approximate grab sampling period can be determined for the site specific field conditions, which would be used for subsequent sampling locations,
- > Once sampling is completed and the canister has been removed, install ¼ inch brass screw-on cap onto the SUMMA® canister,
- > Label the canister with the site name, sampling location, date, time, analytical parameter, and sampler identification, and
- > Transport the vapor sample canisters to certified laboratory via overnight courier using proper chain-of-custody documentation.

## 3.2.2 Vapor Sample Collection using NIOSH Tubes

NIOSH tube sampling is needed to determine the concentration of TPH (diesel range) in the vapor samples. The NIOSH tube contains an activated charcoal media. The following steps will be followed for obtaining vapor samples using NIOSH Tubes:

- The NIOSH tubes should be received from the laboratory in a cooler with an inside temperature of 2 to 6 degrees Celsius using "Blue Ice" or equivalent;
- A GilAir personal sampling pump is needed to obtain the vapor samples. The pump will be calibrated prior to use;
- The sampling tubing is connected to the NIOSH tube;
- The sample is collected by connecting the opposite end of the NIOSH tube to a personal sampling pump and drawing the soil gas through the tube as described in the sampling method for Naphthas 1550 (Appendix E). Documentation of flow rate and sampling time will be entered in the field notebook, as it will be needed to calculate the sample concentration;
- The pump is then turned off and the NIOSH tube is disconnected from the pump and tubing, the ends of the tube are capped, and the tube is stored for shipping to the laboratory in a cooler with an inside temperature of 2 to 6 degrees Celsius; and
- The tube will be labeled and transported to the certified laboratory as described above for the SUMMA® canister sampling.

### 3.3 Field Documentation

At a minimum, the following information will be recorded in the field logbook for each soil vapor sample collected:



- Ambient air temperature;
- Sample ID will consist of the previous Soil Boring Location ID and SV 01-10.5. This will designate it as a soil vapor sample collected at a depth of 10.5 feet at that previous soil boring ID location. The depth will be rounded to the nearest 0.5 foot;
- Sample depth (with calculations if boring is performed at an angle);
- Sample location with schematic showing at least two measurements to fixed site features,
- Sample date (e.g. mm/dd/yy 09/21/02);
- Sample time in 24-hour format (e.g. 13:50 for 1:50 in the afternoon);
- Length of sampling time;
- · Beginning and ending canister pressure; and
- For NIOSH tube sampling, the flow rate and length of sampling time.

## 3.4 Soil Chemistry and Geotechnical Parameter Samples

Subsurface soil samples will be collected continuously from the surface to the capillary fringe (determined by the field Geologist) in an adjacent boring to those used to obtain soil vapor samples using a Geoprobe Macro Sampler. A soil sample from each boring corresponding to the depth of the vapor sampling location will be split, and half analyzed for chemical parameters and the other half analyzed for geotechnical parameters to assist in the evaluation of the soil vapor results.

Soil samples from Areas 2B and 6C will be analyzed for VOCs, MTBE, naphthalene, TPH-GRO (gasoline range), and TPH-DRO (diesel range). At Areas 3C and 3G, the soil samples will be analyzed for BTEX, MTBE, naphthalene, TPH-GRO, and TPH-DRO.

The split samples will be analyzed for the following geotechnical parameters; Volumetric Water/Moisture Content, and Fractional Organic Carbon.

## 4.0 Laboratory Analysis Procedures

The SUMMA® canisters and NIOSH tubes will be prepared for sampling by the certified laboratory selected for laboratory analysis. Prior to sampling, each canister will be cleaned and blanked. Following cleaning and blanking, the canisters will be evacuated, leak-checked, their vacuum measured, and prepared for field deployment.

The soil vapor and background air samples will be analyzed for selected VOCs as specified below following guidance outlined in Environmental Protection Agency (EPA) Methods Modified TO-15 and TO-3, and NIOSH Method 1550.

- BTEX and MTBE Modified TO-15 (Std. Level),
- Naphthalene Modified TO-15+Naphthalene (Std. Level),
- TPH gasoline range (C5-C10 or C2-C12) Modified TO-3 (Std. Level),
- TPH-D (diesel range) NIOSH 1550, and
- 2-propanol (leak test) Modified TO-15 (Std. Level).

# 5.0 Quality Assurance – Quality Control Procedures

Quality Control (QC) on the project will consist of method appropriate laboratory quality control measures run to evaluate the precision and accuracy of the analytical methods. QC for the project field work will consist of performing and documenting equipment/instrument specific calibrations, inspections, and tests to evaluate the equipment precision and accuracy. Additionally, equipment that does not perform to specifications or is defective is taken out of service, clearly identified, and segregated until it has been repaired and shown by calibration/verification to perform satisfactorily. Routine maintenance is performed as needed, depending on how often the equipment/instrument is used, the manufacturer's recommendations, and previous experience.

Quality Assurance (QA) for the project will consist of review of the field data; laboratory data; evaluation of QC problems; coordination with the laboratory or field personnel to answer questions and resolve problems; and technical review of the report.

## 6.0 Reporting

Following completion of the TPH soil vapor sampling, a report will be completed that documents the vapor sampling investigation results including a summary of site activities and laboratory analysis. Copies of the laboratory reports and chain-of-custody forms will also be included in the report. Analysis of the data will be used to supplement the risk assessment already submitted to the Airport and MDNR following the protocol included in the MRBCA Guidance Document.

## 7.0 References

- MACTEC Engineering and Consulting, Inc. (MACTEC). 2004. RCRA Facility Investigation Report for McDonnell Douglas, Hazelwood, Missouri.
- MACTEC. 2003. Environmental Field Investigation for Boeing Tract 1 South Property. Hazelwood, Missouri Facility.
- Risk Assessment & Management (RAM) Group, Inc. 2004. Risk Based Corrective Action Report for McDonnell Douglas, Hazelwood, Missouri.
- RAM Group, Inc. 2004a. Standard Operating Procedures (SOP) for Soil Vapor Grab Sampling Activities Using the Geoprobe® Post-Run Tubing System With 6-Liter SUMMA® Canisters and NIOSH Tubes.

**Tables** 

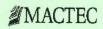


Table 1-1 List of Constituents of Concern for Risk Sub-Areas

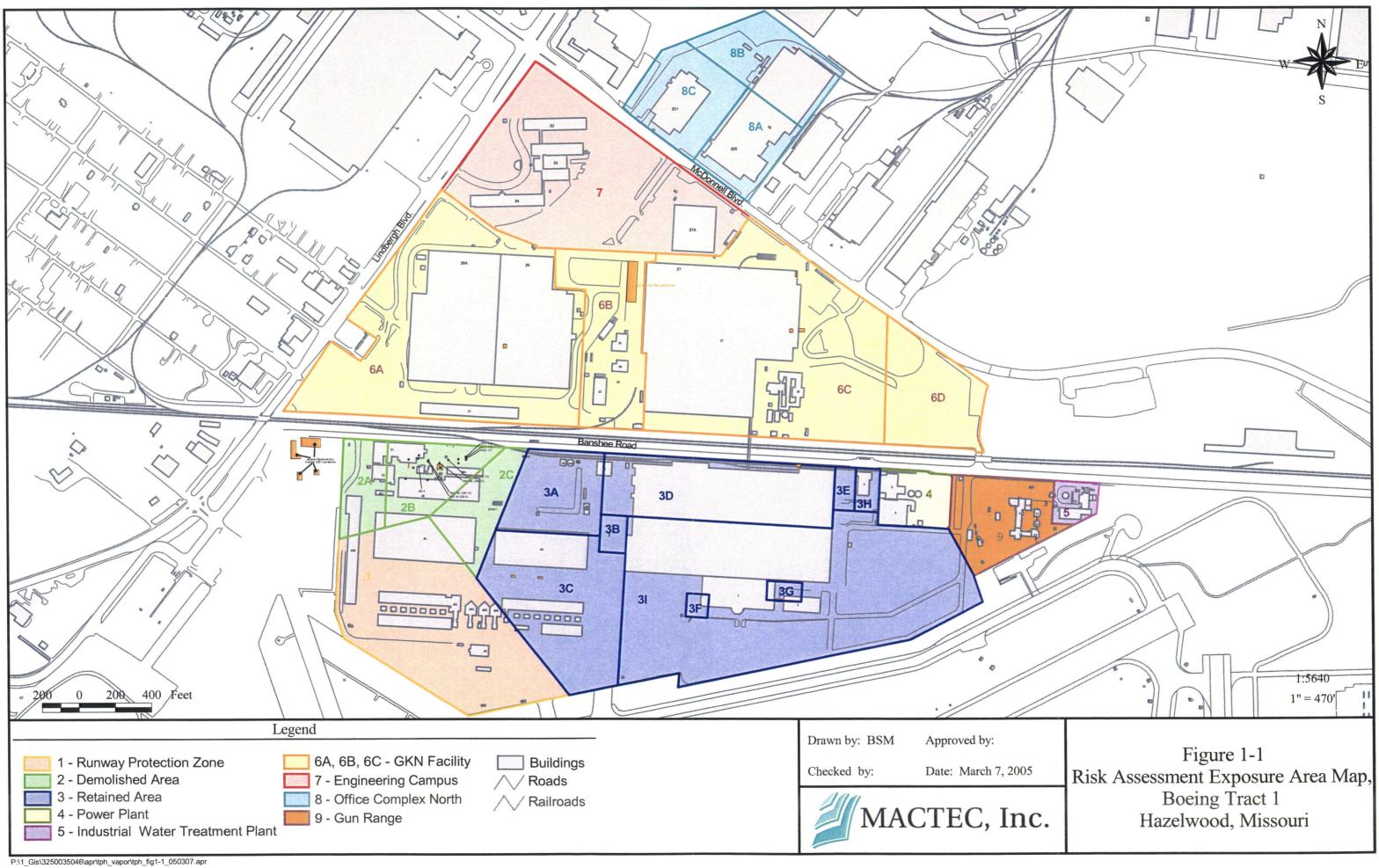
Risk Süb-Area	Avirityies
,	Benzene
,	Ethylbenzene
,	Methyl-tert-butyl ether
All	Naphthalene
7311	2-Propanol
	Toluene
	Xylenes
	TPH
	Cis-1,2-Dichloroethene
	1,1-Dichloroethene
	n-Butylbenzene
	n-Propylbenzene
	Sec-Butylbenzene
2B	Tetrachloroethene
	Trans-1,2-Dichloroethene
	Trichloroethene
	1,2,3-Trimethylbenzene
	1,2,4-Trimethylbenzene
	Vinyl Chloride
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	Cis-1,2-Dichloroethene
6C	2-Hexanone
	Trichloroethene
	Vinyl Chloride

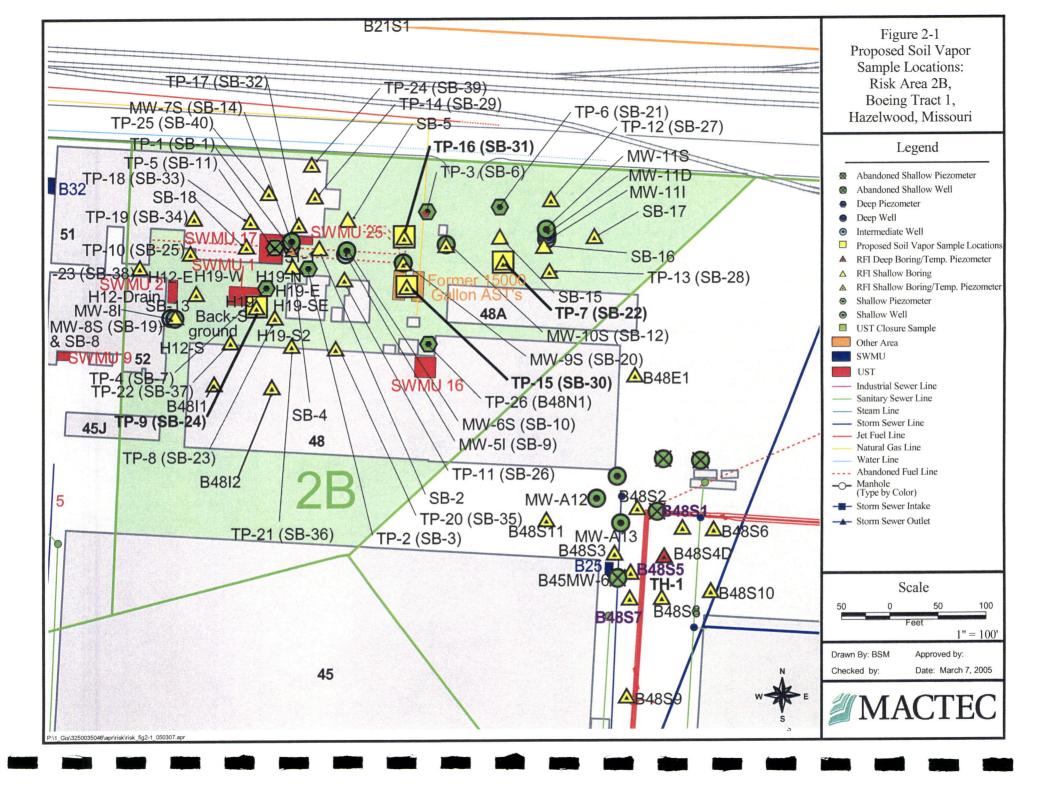
Table 2-1 Historical Depth to Groundwater Measurements, Boeing Tract 1, Hazelwood, Missouri

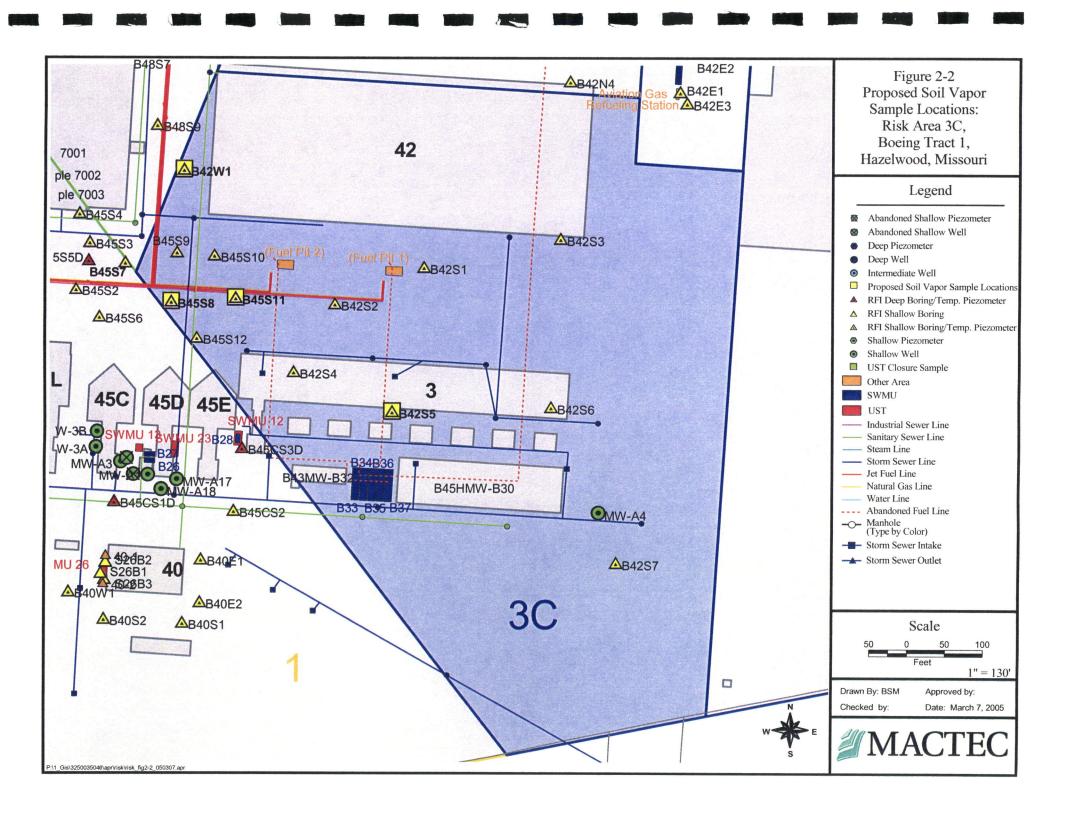
Risk Area	Well ID	Date	Depth to Water (feet)
	T T	2/20/2001	
l		7/25/2001	
		10/29/2001	7.57
1		12/17/2001	5.39
		3/5/2002	6.58
		6/3/2002	
	MW-11S	8/13/2002	
1	j	8/16/2002	6.35
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		3/12/2003	5.61
		3/21/2003 6/23/2003	7.46 7.44
2B	<del> </del>	9/28/2000	5.83
		10/5/2000	5.87
		2/21/2001	4.79
		7/23/2001	5.15
		7/27/2001	4.29
	1	10/30/2001	4.37
	MW-9S	12/19/2001	3.91
	1	3/5/2002	4.75
		5/30/2002	4.06
		8/16/2002	4.79
		12/17/2002	7.06
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	B25MW1	7/23/2001	9.70
		10/24/2001	9.08
		3/11/2002	9.42
		2/21/2022	0.04
		3/21/2002	9.34
	·	5/31/2002	10.93
		5/31/2002 7/28/2000	10.93 9.34
		5/31/2002 7/28/2000 10/5/2000	10.93 9.34 9.10
	MW5	5/31/2002 7/28/2000 10/5/2000 1/5/2001	9.34 9.10 9.51
	MW5	5/31/2002 7/28/2000 10/5/2000 1/5/2001 1/11/2001	10.93 9.34 9.10 9.51 9.45
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6C	MW5	5/31/2002 7/28/2000 10/5/2000 1/5/2001 1/11/2001 5/7/2001 7/24/2001 7/28/2001	10.93 9.34 9.10 9.51 9.45 9.53 9.35 9.35
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6C		5/31/2002 7/28/2000 10/5/2000 1/5/2001 1/11/2001 5/7/2001 7/28/2001 1/5/2001 1/8/2001 5/7/2001 7/19/2001 10/25/2001 3/11/2002 5/31/2002 8/16/2002	10.93 9.34 9.10 9.51 9.45 9.53 9.35 9.35 8.18 8.06 8.48 7.94 7.39 7.91 8.16 8.26
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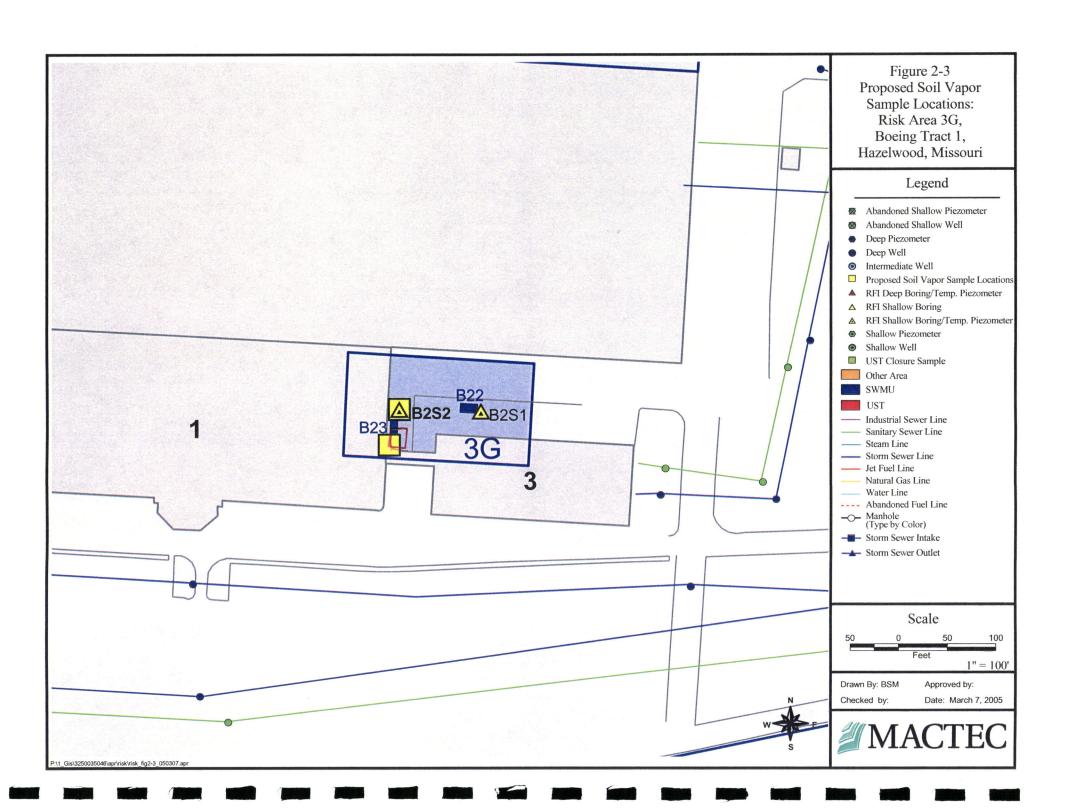
**Figures** 

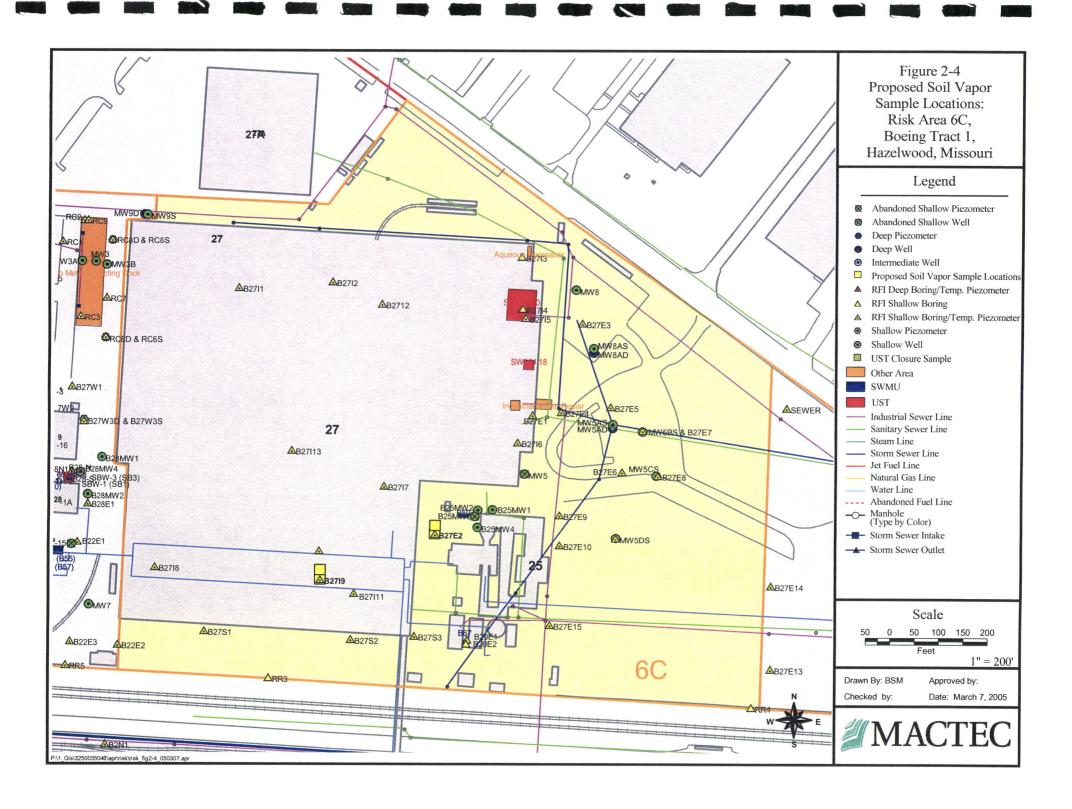






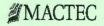


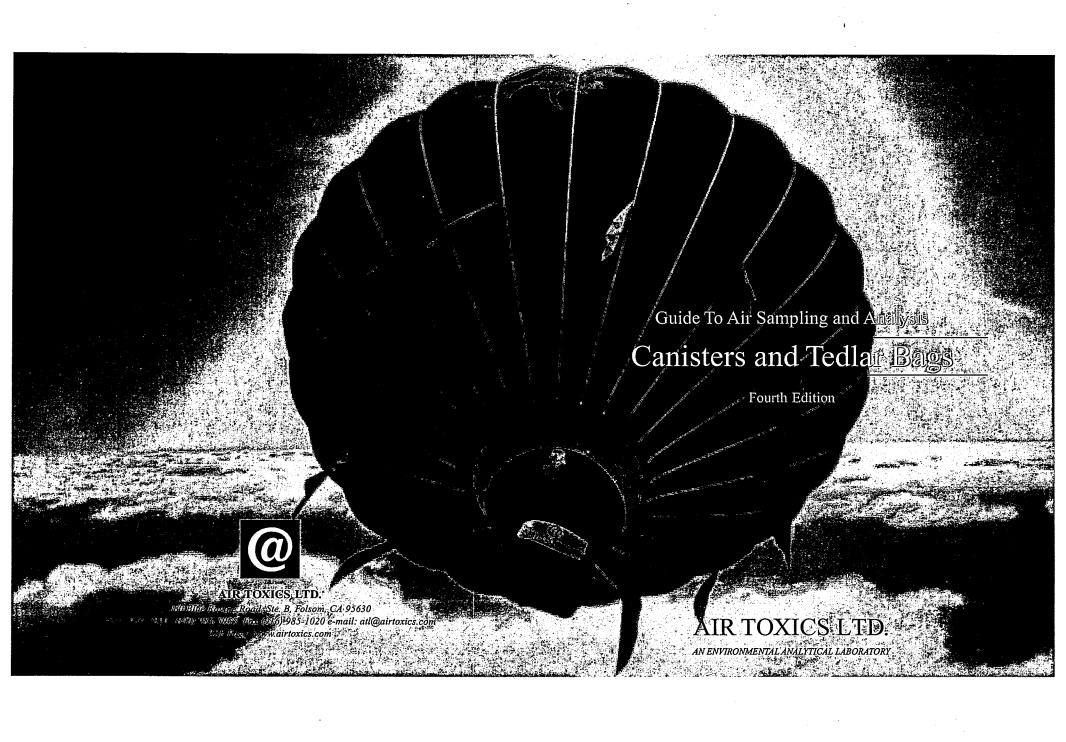




# Appendix A

Guide to Air Sampling and Analysis, Canisters and Tedlar® Bags, Fourth Edition, Air Toxics Ltd









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- 4.2 Considerations for Sampling at Altitude
- Considerations for Soil/Landfill Gas Sampling
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- 1.2 Comparison of Canisters to Tedlar Bags
- 2.2.3 Fill Times for Canisters
- 2.4.3 Flow Rates for Selected Sampling Intervals
- 2.4.4 Relationship Between Final Canister Vacuum, Volume Sampled, and Dilution Factor
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#### Section 1.0 Introduction

Air Toxics Ltd. presents this guide as a resource for individuals engaged in air sampling. Air sampling can be more involved than water or soil sampling due to the reactivity of chemical compounds in the gas matrix and sample interaction with the sampling equipment and media. Ensuring that air samples are collected properly is an important step in acquiring meaningful analytical results. This guide is not a substitute for experience and cannot possibly address the multitude of actual field conditions. Note that this guide is intended for typical projects involving whole air sampling of volatile organic compounds (VOCs) in canisters and Tedlar bags. Air Toxics Ltd. provides the "Guide to Air Sampling and Analysis - Sorbents, Solutions, and Filters" for other types of sampling.

#### 1.1 Whole Air Sampling of VOCs

There are four general ways to collect compounds in a gas phase sample. A sampler can collect the gas in a container or draw the gas through a sorbent, solution, or filter. This guide focuses on collecting a sample in the most common air sampling containers, Summa canisters and Tedlar bags. The sample can be collected in the container either passively (i.e., by evacuating the canister prior to sampling) or actively (i.e., using a pump). The container is subsequently sealed and transported to the laboratory for analysis. The sample is referred to as a "whole air sample" and the compounds remain in the gas matrix (e.g., ambient air) inside the container.

As a general rule, whole air sampling is best when target compounds are volatile, non-polar, and have boiling points less than 170°C, although exceptions to this rule can be found. Recovery of any given compound in a whole air sample is very much dependent upon the humidity of the sample, the chemical activity of the sample matrix, and the degree of inertness of the container.

#### 1.2 Choosing Between Canisters and Tedlar Bags

Deciding whether a canister or a Tedlar bag should be used for collecting a whole air sample depends on the type of air sampling application. The Tedlar bag is best used as a "ppmv" (parts per million by volume) whole air sample container. In other words, it is best suited for air sampling applications involving compound concentrations well above the low ppbv (parts per billion by volume) range. Soil/landfill gas surveys, monitoring soil vapor extraction (SVE) systems, and sampling for atmospheric/fixed gases are applications well suited for Tedlar bag sampling. Ambient and indoor air projects driven by risk assessment or litigation are better suited for Summa canisters that are cleaned and individually certified free of the target compounds. The different degree of compound inertness between the two sample container surfaces is reflected in their suggested hold times for VOCs – 3 days from sampling to analysis for a Tedlar bag compared to 14-30 days for a Summa canister. Analyses of new Tedlar bags reveal that some VOCs may be present at concentrations in the single digit ppbv range (see Section 3).

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**Table 1.2. Comparison of Canisters to Tedlar Bags** 

	Canisters	Tedlar Bags
Common Volumes	∀ <b>1 and 6 L</b> ুলিক ন্যুক্তিকক	≴ 1, 3, and 5 L
Type of Sampling	Passive (vacuum)	Active (pump required)
Sample Handling	Room temperature	Room temperature
Media Hold Time	Up to 30 days recommended	Indefinite
Hold Time to Analysis	14-30 days	3 days
Surface Inertness	Excellent	Fair - Gradult galif
Cleanliness	10% or 100% certified to ppbv/pptv levels	Some VOCs present at 0.5 to 45 ppbv
Sampling Application	Ambient/indoor air, soil/landfill gas, stationary source	Ambient air (fixed gases only), soil/landfill gas, stationary source
Rule of Thumb	"ppby device"	"ppmv device"
Advantages	Inertness, hold time, ruggedness, no pump	Purchase/shipping cost, availability, convenience

The table above compares the features of canisters and Tedlar bags. Canisters have superior inertness, hold time to analysis, ruggedness, and do not require a sampling pump. Tedlar bags can be purchased inexpensively in bulk, carried to a sampling site in a briefcase, filled in seconds, and shipped easily to the laboratory for analysis. Call Client Services at 800-985-5955 if you have questions regarding sampling media.

#### 1.3 Organization of this Guide

The remainder of this guide is divided into three sections: canister sampling, Tedlar bag sampling, and special sampling considerations. Section 2 on canister sampling and Section 3 on Tedlar bag sampling provide complete sampling media descriptions, practical considerations for sampling, and step-by-step sampling procedures. Photographs illustrate the correct way to assemble the various sampling components. Tables provide detailed information on many operational factors that ultimately influence the quality of the data obtained from a canister or Tedlar bag sample. Section 4 provides considerations for special sampling configurations such as field duplicates and ambient blanks. This section also provides considerations for sampling at altitude, soil/landfill gas sampling, and sample cylinder (or "sample bomb") sampling.

If you have any questions after reading this guide, please call Client Services at 800-985-5955 before proceeding with sampling. Air Toxics Ltd. also provides technical articles on specific air topics in Air Topics publications and In the Air quarterly newsletters available upon request or on the Internet at www.airtoxics.com.

## Section 2. Canister Sampling

This section provides a description of air sampling canisters, practical considerations for sampling, and step-by-step instructions for collecting a grab and integrated sample. Photographs illustrate the correct way to assemble the various sampling components. Tables provide detailed information on many operational factors that ultimately influence the quality of the data obtained from a canister sample.

#### 2.1 Introduction to Canisters

An air sampling canister is a container for collecting a whole air sample for ambient and indoor air applications. The canister is best suited for projects involv-

ing analysis of compounds in the ppbv range. However, canisters can be used for other applications such as landfill and soil gas involving analysis of compounds in the ppmv range.

A canister can be spherical or cylindrical and is constructed of stainless steel. The canister is prepared for sampling by evacuating the contents to a vacuum of approximately 29.9 inches of Mercury (in. Hg). Opening the stainless steel bellows valve allows the air sample to enter the canister. When the target volume of sample is collected, the valve is closed and the canister is returned to the laboratory.



Canisters can range in volume from less than 1 liter (L) to greater than 6 L. At Air Toxics Ltd., 6 L canisters are used for ambient air samples and for taking integrated samples. 1 L canisters are normally used for taking high concentration (i.e., greater than 5 ppbv) grab samples, although exceptions to these guidelines are common. Variations of air sampling canisters include glass bulbs, sample cylinders (or "sample bombs"), and Summa canisters. Glass bulbs are rarely used in field applications due to lack of ruggedness. Sample cylinders are DOT-approved, high pressure, thick-walled, stainless steel cylinders with a valve at each end (see Section 4.4). The remainder of this section focuses on Summa canisters.



#### 2.1.1 Summa Canister

A Summa canister is a stainless steel container that has had the internal surfaces specially passivated using a "Summa" process. This process combines an electropolishing step with a chemical deactivation step to produce a surface that is nearly chemically inert. A Summa surface has the appearance of a mirror: bright, shiny, and smooth. The degree of chemical inertness of a whole air sample container is crucial to minimizing reactions with the sample and maximizing recovery of target compounds from the container. Air Toxics Ltd. maintains a large inventory of Summa canisters in 6 and 1 L volumes.

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#### 2.1.2 Canister Cleaning and Hold Time

Canister sampling differs considerably from collecting a water sample in a VOA vial or a soil sample in an amber jar in that the container (valued at over \$450) is cleaned and reused. A canister will hold a high vacuum (i.e., greater than 25 in. Hg) for more than 30 days. Air Toxics Ltd., however, requires that our canisters be returned within 30 days.

#### Media hold time for a canister is 30 days

Air Toxics Ltd. provides two types of canister cleaning certification, 10% and 100%, depending upon the requirements of the project. The 10% certification process is appropriate for routine ambient air applications and high concentration applications such as soil vapor and landfill gas monitoring. The 10% certification process begins by cleaning canisters using a combination of dilution, heat, and high vacuum. After completing the cleaning steps, 10% of the canisters are certified each day. Canisters are



certified for approximately 60 VOCs using GC/MS by Modified EPA Method TO-15. The 10% certification process requires that target compound concentrations be below 0.2 ppbv. Alternatively, the 100% certification (i.e., individual certification) process is appropriate for ambient and indoor air applications driven by risk assessment or litigation that require pptv (parts per trillion by volume) sensitivity. Similar to the 10% certification, the 100% certification also begins with the canister cleaning process. The difference with the 100% certification is that canisters are individually certified for a client-specific list of target compounds using GC/MS by TO-15. The 100% certified canisters are shipped with analytical documentation demonstrating that they are free of the target compounds down to the project reporting limits.

# Specify whether your project requires 10% or 100% canister cleaning certification

Although 14 days is the most commonly cited hold time for a canister sample, the hold time is compound specific. For example, non-polar compounds such as chloroform, benzene, and vinyl chloride are stable in a canister for at least 30 days. In fact, EPA Method TO-15 states: "Fortunately, under conditions of normal usage for sampling ambient air, most VOCs can be recovered from canisters near their original concentrations for after storage times of up to thirty days". However, recovery of polar compounds such as methanol and acetone begin to drop significantly after 14 days. Analysis of these samples should be performed within 14 days.

Sample hold time to analysis for a canister is 14-30 days for VOCs

#### 2.2 Associated Canister Hardware

Associated hardware used with the canister includes the valve, brass cap, particulate filter, and vacuum gauge.

#### 2.2.1 Valve

An industry standard, 1/4 in. stainless steel bellows valve (manufactured by Nupro) is mounted at the top of the canister. The valve allows vacuum to be maintained in the canister prior to sampling and seals off the canister once the sample has been collected. No more than a half turn by hand is required to open the valve. Do not over-tighten the valve after sampling or it may become damaged. A damaged valve can leak and possibly compromise the sample. Some canisters have a metal cage near the top to protect the valve.

#### 2.2.2 Brass Cap

Each canister comes with a brass cap (i.e., Swagelok 1/4 in. plug) secured to the inlet of the valve assembly. The cap serves two purposes. First, it ensures that there is no loss of vacuum due to a leaky valve or valve that is accidentally opened during handling. Second, it prevents dust and other particulate matter from fouling the valve. The cap is removed prior to sampling and replaced following sample collection.

Always replace the brass cap following canister sampling





7 Micron

5 Micron

#### 2.2.3 Particulate Filter

Each canister comes with a particulate filter provided separately in the packing box. The filter prevents particulate matter from fouling the valve (or flow controller) and entering the canister. Particulate filters should be cleaned between uses. Air Toxics Ltd. provides two types of particulate filters: 7 micron and 5 micron. The longer, 7 micron particulate filter is normally used with 6 L canisters and whenever an integrated sample is being collected. This device filters particulate matter greater than 7 microns in diameter and does not significantly restrict the flow rate in to the canister. Typical fill times for canisters are shown in the following table. The shorter, 5 micron particulate filter is often used to slow down grab sampling with 1 L canisters and mini-cans. This device is a fritted stainless steel disk that has been pressed into a conventional Swagelok adapter. This device filters particulate matter greater than 5 microns in diameter and has a relatively high pressure drop across the fritted disk. It restricts the flow into the canister and fill times are increased.

Always use the particulate filter for canister sampling

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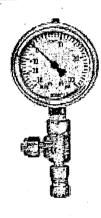
#### **Table 2.2.3 Fill Times for Canisters**

CANISTER VOLUME	7 micron filter	5 micron filter
6 L	16 sec	23 min
1 L	3 sec	4 min
400 mL (mini-can)	1-2 sec	1 min 20 sec

#### 2.2.4 Vacuum Gauge

A vacuum gauge can be used to measure the initial vacuum of the canister before sampling and the final vacuum upon completion. A gauge can also be used to monitor the fill rate of the canister when collecting an integrated sample. Gauges are generally not used during the brief interval for grab sampling. Gauges are used only to provide a relative measure of "change". The accuracy of gauges provided by Air Toxics Ltd. is such that gauge-to-gauge comparisons have no merit. Individuals engaged in frequent air sampling or air projects driven by risk assessment or litigation are highly encouraged to purchase and maintain their own gauge. Upon request, Air Toxics Ltd. provides two types of gauges: vacuum gauges reading 0 to 30 in. Hg and vacuum-pressure gauges reading 30 in. Hg to 30 psig (pounds per square inch gage).



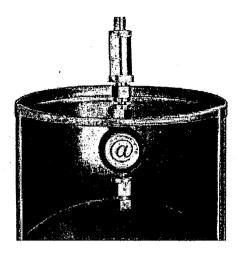


#### Air Toxics Ltd. provides gauges only if requested

## 2.3 Grab Sampling with Canisters

There are two basic modes of canister sampling: grab and integrated. A grab sample is taken over a short interval (i.e., 1-5 minutes) while an integrated sample is taken over an extended period (e.g., 0.5-2 hours for a 1 L canister and 0.5-24 hours for a 6 L canister). In both modes the canister vacuum is used to draw sample into the canister. This is commonly referred to as passive sampling. Active sampling utilizes a pump to fill the canister. The most common hardware configuration used to take a grab sample are illustrated in the following figure. A particulate filter is used to prevent particulate matter from fouling the valve and entering the canister.







#### 2.3.1 Considerations for Grab Sampling With Canisters

The following are some considerations for collecting a grab sample in a canister.

- Avoid Leaks in Sampling Train: All fittings on the sampling hardware are 1/4 in. Swagelok. A
  9/16 in. crescent wrench is used to assemble the hardware. It is not necessary to over tighten the
  fittings; finger tight plus 1/4 turn with the wrench is adequate. In practice this should be tight
  enough so that the various pieces of equipment, when assembled, cannot be rotated by hand.
- Verify Gauge Operation: If the indicator does not read "zero" upon arrival, the gauge either
  needs to equilibrated or the gauge may be damaged and unusable. Equilibrate the gauge by
  "cracking" the rubber plug on top of the gauge. For more details on the equilibration procedure,
  see instructions included with the gauge or call Client Services at 800-985-5955.
- Verify Initial Vacuum of Canister: Prior to shipment, each canister is checked for mechanical
  integrity. However, it is still important to check the vacuum of the canister prior to use and record
  the initial vacuum on the chain-of-custody. The initial vacuum of the canister should be greater
  than 25 in. Hg. If the canister vacuum is less than 25 in. Hg, do not use it. Call Client Services at
  800-985-5955 and arrange for a replacement canister. If sampling at altitude, there are special
  considerations for gauge readings and sampling (see Section 4.2). The procedure to verify the
  initial vacuum of a canister is simple, but unforgiving.

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- 1. Confirm that valve is closed (knob should already be tightened clockwise)
- 2. Remove the brass cap
- 3. Attach gauge
- 4. Attach brass cap to side of gauge tee fitting
- 5. Open and close valve quickly (a few seconds)
- 6. Read vacuum on the gauge
- 7. Record gauge reading on "Initial Vacuum" column of chain-of-custody
- 8. Verify that canister valve is closed and remove gauge
- 9. Replace the brass cap
- Leave Residual Vacuum: A grab sample can be collected either by allowing the canister to reach
  ambient conditions or by leaving some residual vacuum (e.g., 5 in. Hg) in the canister. In either
  case, the final vacuum should be noted on the "Final Vacuum" column on the chain-of-custody.
  This will enable the laboratory to compare the final vacuum with the receipt vacuum (i.e., the
  vacuum measured upon arrival at the laboratory). If the two readings differ significantly, Client
  Services will contact you for instructions on how to proceed.

#### 2.3.2 Step-by-Step Procedures for Canister Grab Sampling

These procedures are for a typical ambient air sampling application and actual field conditions and procedures may vary.

#### Before you get to the field:

- Verify contents of the shipped package (e.g., chain-of-custody, canister, particulate filter, and gauge – if requested)
- Verify that gauge is working properly (see Section 2.3.1)
- 3. Verify and record initial vacuum of canister (see Section 2.3.1)

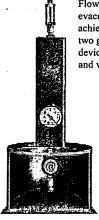
#### When ready to sample:

- 4. Remove brass cap
- 5. Attach particulate filter to canister
- 6. Open valve 1/2 turn (6 L canister normally takes about 16 sec to fill)
- 7. Close valve by hand tightening knob clockwise
- 8. Verify and record final vacuum of canister (repeat steps used to verify initial vacuum)
- 9. Replace brass cap
- 10. Fill out canister sample tag
- 11. Return canister in box provided
  - Unreturned canister charge of \$450 each
- 12. Return sample media in packaging provided. Unreturned equipment charges:
  - \$45 per particulate filter
  - \$45 per gauge
- 13. Fill out chain-of-custody and relinquish samples properly
- 14. Place chain-of-custody in box and retain pink copy
- 15. Tape box shut and affix custody seal (if applicable) across flap
- 16. Ship accordingly to meet method holding times

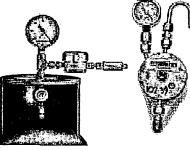


#### 2.4 Integrated Sampling with Canisters and Flow Controllers

An air sample collected over more than a few minutes is referred to as an integrated sample and can provide information on compound concentrations in air averaged or composited over time. An 8- or 10-hour integrated sample can be used to determine indoor air quality in the workplace. Similarly, a 24-hour integrated sample can be an economical and practical approach to determine residential exposure to indoor or outdoor air sources. The most common hardware configurations used to take an integrated sample are illustrated below.



Flow controllers are devices that regulate the flow of air during sampling into an evacuated canister. Also known as flow restrictors, these devices enable a sampler to achieve a desired flow rate and thus, a sampling interval. Air Toxics Ltd. provides two general types of flow controllers: mass flow controllers and critical orifice devices. Both devices are driven by differential pressure between ambient conditions and vacuum in the canister.



Flow controller by



Flow controller by Veriflo

#### 2.4.1 Mass Flow Controller

A mass flow controller employs a diaphragm that actively compensates to maintain a constant mass flow rate. As the differential pressure decreases, the flow rate tends to decrease and the diaphragm responds by opening up to allow more air to pass through. Mass flow controllers can be adjustable or fixed and can provide integrated samples with intervals ranging from hours to days. Air Toxics Ltd. provides a fixed mass flow controller that is calibrated at the laboratory for 24-hour sampling. Adjustable mass flow controllers have a knob that can be adjusted in the field to provide integrated samples with intervals ranging from one to 24 hours. The rugged conditions of field sampling are not usually compatible with adjustable mass flow controllers and Air Toxics Ltd. designed a more reliable flow controller based on a critical orifice design.

#### 2.4.2 Critical Orifice Device

Air Toxics Ltd. designed a critical orifice flow restrictor to provide integrated samples with intervals from 0.5 to 8 hours. The device restricts air flow by forcing the sample to enter a capillary column of minute radius. This device is passive compared to an actively compensating diaphragm and the flow rate decreases as the driving force (differential pressure) decreases. For sampling intervals from 0.5 to

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8 hours, however, the flow rate is relatively constant. The main advantages of the Air Toxics Ltd. flow restrictors are improved ruggedness and cleanliness. With no moving or adjustable parts, the Air Toxics Ltd. design is unlikely to lose its flow setting. In addition, a vacuum gauge is built in to the device to monitor sampling progress. To ensure there are no contamination issues from previous use, the capillary column is replaced before shipping to the field.



#### 2.4.3 Sampling Interval and Flow Controller Setting

When you request canisters and flow controllers from Air Toxics Ltd., you will be asked for the sampling interval, and the flow controllers will be pre-set prior to shipment according to the table below. The flow controller is set to collect 5 L of sample over the sample interval. Final canister vacuum is targeted at 5 in. Hg. The flow rate is set at standard atmospheric conditions (approximately sea level). If the air sample is a process (pressurized or under vacuum) or is collected at elevation, the canisters will fill faster or slower depending on the sampling conditions. If you specify the pressure of the source at project set-up, we can set the flow controller accordingly. See Section 4 for a discussion of collecting a sample at elevation. The 24-hr flow controllers should not be used for process or source samples.

Table 2.4.3 Flow Rates for Selected Sampling Intervals (mL/min)

Sampling Interval (hrs)	0.5	1	2	4	8	12	24
6 L Canister	167	83.3	41.7	20.8	11.5	7.6	3.5
1 L Canister	26.6	13.3	6.7	-	-	-	

Note: Target fill volumes for 6 L and 1 L canisters are 5,000 mL and 800 mL, respectively.

Flow Rate(mL/min) = Target Fill Volume (mL)
Sampling Interval (min)

#### 2.4.4 Final Canister Vacuum and Flow Controller Performance

Ideally the final vacuum of a 6 L canister should be 5 in. Hg or greater. As long as the differential pressure is greater than 4 in. Hg ambient pressure, then the flow through the device will remain approximately constant as the canister fills. If there is insufficient differential pressure, the flow through the controller will decrease as the canister pressure approaches ambient. Because of the normal fluctuations in the flow rate (due to changes in ambient temperature, pressure, and diaphragm instabilities) during sampling, the final vacuum will range between 2 and 10 in. Hg.

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- If the residual canister vacuum is greater than 5 in. Hg (i.e., more vacuum), the flow rate was low and less than 5 L of sample was collected. When the canister is pressurized to 5 psig prior to analysis, sample dilution will be greater than normal. This will result in elevated reporting limits.
- If the residual canister vacuum is less than 5 in. Hg (i.e., less vacuum), the initial flow rate was
  high. Once the vacuum decreases below 5 in. Hg, the flow rate begins to drop significantly. This
  scenario indicates that the sample is skewed in favor of the first portion of the sampling interval.
- If the final vacuum is near ambient (i.e., less than 1 in. Hg), there is inadequate differential pressure to drive the flow controller. The sampler cannot be certain the desired sampling interval was achieved before the canister arrived at ambient conditions. The sample could have been acquired over a 1-hour interval (which would be the case if the connection between the canister and flow controller leaked or if the flow controller malfunctioned) or a 24-hour interval. Although the actual sampling interval is uncertain, the canister still contains sample from the site.

Table 2.4.4 Relationship Between Final Canister Vacuum, Volume Sampled, and Dilution Factor (6 L Canister)

Final Vacuum (in. Hg)	0	2.5	5	7.5	10	12.5	15	17.5	20	
Volume Sampled (L)	6	5.5	5	4.5	4	3.5	. 3	2.5	2	
Dilution Factor*	1.34	1.46	1.61	1.79	2.01	2.30	2.68	3.22	4.02	

\* Canister pressurized to 5 psig for analysis

#### 2.4.5 Considerations for Integrated Sampling with Canisters

Collecting an integrated air sample is more involved than collecting a grab sample. Sampling considerations include verifying that the media is ready, monitoring the integrated sampling progress, and avoiding contamination.

Avoid Leaks in the Sampling Train: See Section 2.3.1 for instructions on how to securely
assemble sampling hardware. A leak in any one of these connections means that some air will be
pulled in through the leak and not through the flow controller. A final pressure near ambient is one
indication that there may have been a leak.

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- Verify Initial Vacuum of Canister: See Section 2.3.1 for instructions on verifying initial canister
  vacuum. If you are using an Air Toxics Ltd. critical orifice flow controller, note that you can use
  the built-in gauge. It is important to note both the canister and flow controller serial numbers on
  the chain-of-custody.
- Monitor Integrated Sampling Progress: It is a good idea to monitor the progress of the integrated sampling during the sampling interval. The volume of air sampled is a linear function of canister vacuum. For example, halfway (4 hours) into an 8-hour sampling interval, the canister should be half filled (2.5 L) and the gauge should read approximately 17 in. Hg. More vacuum than 17 in. Hg indicates that the canister is filling too slowly; less than 17 in. Hg and the canister is filling too quickly. If the canister is filling too slowly, a valid sample can still be collected (see Section 2.4.4). If the canister is filling too quickly because of a leak or incorrect flow controller setting, corrective action can be taken. Ensuring all connections are tight may eliminate a leak. It is possible to take an intermittent sample. The time interval need not be continuous. Eight 1-hour increments, taken by opening and closing the canister valve, will yield a valid sample.

Table 2.4.5 Gauge Readings for an 8-Hour Sampling Interval

Sampling Interval (hrs)	0	4	8	
Canister Vacuum (in. Hg)	29.9	17.4	5	
Volume Sampled (L)	0	2.5	5	

- Avoid Contamination: Flow controllers should be cleaned between uses. This is normally accomplished by returning them to the laboratory. For large air sampling projects, Air Toxics Ltd. has designed a field conditioning program for 24-hour flow controllers involving a purge manifold. This arrangement provides the sampler with scheduling flexibility, inventory control, and convenience in the field. Air Toxics Ltd. will provide the 24-hour flow controllers, a purge manifold, Teflon tubing, rubber ferrules, vacuum pump, and flow meter. The sampler will need to provide the certified nitrogen cylinder and the certified high pressure regulator. Call Client Services at 800-985-5955 if you are interested in the field conditioning program.
- Keep Sampling Train Out of Direct Sunlight: The sampling train should be kept out of direct sunlight during sampling. There will be some flow rate drift if the temperature of the controllers is allowed to vary significantly.

#### 2.4.6 Step-by-Step Procedures for Integrated Sampling

These procedures are for a typical ambient air sampling application and actual field conditions and procedures may vary.

#### Before you get to the field:

- Verify contents of the shipped package (e.g., chain-of-custody, canister, particulate filter, and flow controller)
- 2. Verify initial vacuum of canister (see Section 2.3.1)

#### When ready to sample:

- 3. Remove brass cap
- 4. Attach flow controller to canister
- 5. Attach particulate filter to flow controller
- 6. Open valve 1/2 turn
- 7. Monitor integrated sampling progress periodically (see Section 2.4.5)

#### At end of sampling interval:

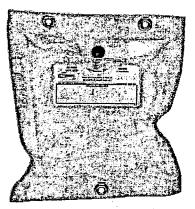
- 8. Verify and record final vacuum of canister (for 24-hr flow controller repeat steps used to verify initial vacuum and for critical orifice device simply read built-in gauge)
- 9. Close valve by hand tightening knob clockwise
- 10. Replace brass cap
- 11. Fill out canister sample tag
- 12. Return canisters in boxes provided
  - Unreturned canister charge of \$450 each
- 13. Return sample media in packaging provided. Unreturned equipment charges:
  - \$45 per particulate filter
  - \$50-500 per flow controller
- 14. Fill out chain-of-custody and relinquish samples properly
- 15. Place chain-of-custody in box and retain pink copy
- 16. Tape box shut and affix custody seal (if applicable) across flap
- 17. Ship accordingly to meet method holding times

## Section 3. Tedlar Bag Sampling

This section provides a description of Tedlar bags, practical considerations for sampling, and step-by-step instructions for collecting a grab sample. Photographs illustrate the correct way to assemble the various sampling components.

### 3.1 Introduction to Tedlar Bags

A Tedlar bag is a container used to collect a whole air sample for landfill gas, soil gas, and stationary source applications. The Tedlar bag is best suited for projects involving analysis of compounds in the ppmv range. However, Tedlar bags can be used for other applications such as ambient air monitoring for atmospheric/fixed gases. They can be used to collect sulfur compounds, but only if the fittings are non-metallic (e.g., polypropylene, Teflon, or Nylon).



A Tedlar bag is made of two plies of Tedlar film sealed together at the edges and features a valve that allows the interior to be filled. Sample collection requires a pressurized sampling port, a low flow rate pump, or a lung sampler. The bag expands as sample enters. When the target volume of sample is collected, the valve is closed and the Tedlar bag is returned to the laboratory. Air Toxics Ltd. maintains a limited inventory of Tedlar bags in 1 L , 3 L, and 5 L volumes.

#### 3.1.1 Tedlar Film

Tedlar is a trade name for polyvinyl fluoride film developed by DuPont Corporation in the 1960's. This patented fluoropolymer has been used in a wide variety of applications including protective surfacing for signs, exterior wall panels, and aircraft interiors. Tedlar film is tough, yet flexible and retains its impressive mechanical properties over a wide range of temperatures (well below freezing to over 200° F). Tedlar exhibits low permeability to gases, good chemical inertness, good weathering resistance, and low off-gassing.

#### 3.1.2 How "Active" is the Surface of a Tedlar Bag?

The surface of a Tedlar bag is a work in progress. The surface of a new bag is essentially free of VOCs at the single digit ppbv level. Compounds detected from analyzing new Tedlar bags include methylene chloride, toluene, acetone, ethanol, and 2-propanol. Note that 2-propanol has been detected in some new bags up to 45 ppbv. Once the Tedlar bag is used, however, the surface has been exposed to moisture and possibly VOCs. It may irreversibly adsorb many VOCs at the low ppbv level. A series of purges with certified gas will not remove the VOCs from the surface. \$15 for a new bag is a small price to pay for peace of mind.

#### Never reuse a Tedlar bag when sampling for trace level compounds

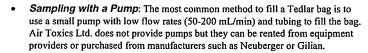
#### 3.1.3 Hold Time for a Tedlar Bag

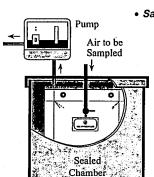
The media hold time for a Tedlar bag is indefinite if stored out of sunlight in a cool, dry location. Tedlar bags can be used to collect samples containing common solvents, hydrocarbons, chlorinated solvents, sulfur compounds, and many other classes of compounds. The sample hold time to analysis varies for different classes of compounds:

- 1 Day: Sulfur compounds (e.g., hydrogen sulfide and methyl mercaptan) and chemically active compounds (e.g., 1,3-butadiene).
- 3 Days: Chlorinated solvents, aromatic compounds, and atmospheric/fixed gases (oxygen, nitrogen, carbon dioxide).

#### 3.2 Tedlar Bag Sampling

Using a Tedlar bag to collect an air sample normally involves "active" sampling, unlike an evacuated canister that can be filled "passively" by simply opening the valve. There are two methods commonly used to fill a Tedlar bag: using a pump or a lung sampler.





• Sampling with a Lung Sampler. Alternatively to using a pump, a "lung sampler" can be used to fill a Tedlar bag.

Although a little more complicated than simply using a pump, the main advantage to using a lung sampler is that it avoids potential pump contamination. A Tedlar bag with attached tubing is placed in a small airtight chamber (even a 5-gallon bucket can work) with the tubing protruding from the chamber. The sealed chamber is then evacuated with a pump causing the bag to expand and drawing the sample through the protruding tube into the bag. The sample air never touches the wetted surfaces of the pump. Air Toxics Ltd. does not provide lung samplers, but they can be rented from equipment suppliers or purchased by manufacturers such as SKC Inc.

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The following are some considerations for collecting a Tedlar bag sample,

- Fill the Tedlar bag no more than 2/3 full: Allow for possible expansion due to an increase in temperature or decrease in atmospheric pressure (e.g., the cargo hold of a plane).
- Keep the Tedlar bag out of sunlight: Tedlar film is transparent to ultraviolet light (although
  opaque versions are available) and the sample should be kept out of sunlight to avoid any photochemical reactions.
- Protect the Tedlar bag: Store and ship the Tedlar bag samples in a protective box at room temperature. An ice chest can be used, but DO NOT CHILL.
- Fill out the Tedlar bag label: It is much easier to write the sample information on the label before
  the Tedlar bag is inflated.
- Provide a second Tedlar bag: Consider filling two bags per location in the rare occasion that a
  defective bag deflates before analysis.
- Avoid Contamination: Care should be taken to avoid contamination introduced by the pump or
  tubing. Begin sampling at locations with the lowest compound concentrations (e.g., sample the
  SVE effluent before the influent). Decontaminate the pump between uses by purging with certified
  air for an extended period; better yet, use a lung sampler. Use shortest length possible of Teflon
  tubing or other inert tubing. Do not reuse tubing. If long lengths of tubing are used, consider
  purging the tubing with several volumes worth before sampling. If you are concerned about
  sampling for trace compounds, you shouldn't be using a Tedlar bag (see Section 1.2).
- Don't Sample Dangerous Compounds in a Tedlar Bag: Do not ship any explosive substances, radiological or biological agents, corrosives, or extremely hazardous materials to Air Toxics Ltd.
   Tedlar bag rupture during transit to the laboratory is possible and the sampler assumes full liability.

#### 3.2.2 Step-by-Step Procedures for Tedlar Bag Sampling (Pump)

Note: These procedures are for a typical stationary source (e.g., SVE system) sampling application; actual field conditions and procedures may vary. See additional sampling considerations in Section 4.3 for sampling soil gas or landfill gas.

#### Before you get to the field:

- Verify contents of the shipped package (e.g., chain-of-custody, Tedlar bag, and tubing/fittings if requested)
- 2. Verify pump cleanliness and operation (Air Toxics Ltd. does not provide pumps)

#### When ready to sample:

- 3. Purge sample port
- 4. Attach new Teflon tubing from sample port or probe to low flow rate pump
- 5. Purge tubing
- 6. Fill out Tedlar bag sample tag
- 7. Attach additional new Teflon tubing from the pump outlet to the Tedlar bag valve
- 8. Open Tedlar bag valve
- 9. Collect sample (FILL NO MORE THAN 2/3 FULL)
- 10. Close Tedlar bag valve by hand tightening valve clockwise
- 11. Return Tedlar bag in boxes provided (DO NOT CHILL)
- 12. Fill out chain-of-custody and relinquish samples properly
- 13. Place chain-of-custody in box and retain pink copy
- 14. Tape box shut and affix custody seal (if applicable) across flap
- Ship priority overnight to meet method holding times. 3 DAY HOLD TIME TO ANALYSIS (most analyses)

## **Section 4. Special Sampling Considerations**

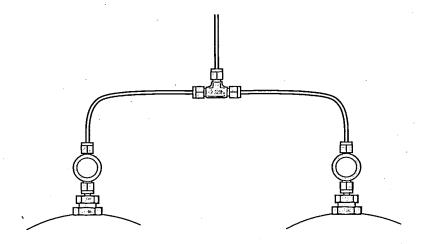
This section provides considerations for special sampling configurations that a sampler may collect in the field such as a field duplicates or an ambient blank. This section also provides considerations for sampling at altitude, soil/landfill gas sampling, and sample cylinder sampling.

#### 4.1 Special Sampling Configurations

Special sampling configurations include a field duplicate, field split, field blank, ambient blank, trip blank, and an equipment rinse. Call Client Services at 800-985-5955 if your project involves any of these special sampling configurations.

#### 4.1.1 Field Duplicate

A field duplicate is a second sample collected in the field simultaneously with the primary sample at one sampling location. The results of the duplicate sample can be compared (e.g., calculate relative percent difference) with the primary sample to provide information on consistency and reproducibility of field sampling procedures. Due to the nature of the gas phase, duplicate samples should be collected from a common inlet. The configuration for collecting a field duplicate includes stainless steel or Teflon tubing connected to a Swagelock "tee". It is imperative that individually certified (i.e., 100% certification process) canisters be used to collect a field duplicate.



#### 4.1.2 Field Split

A field split is similar to a field duplicate in that two samples are collected in the field simultaneously at one sampling location. The main difference is that the samples are sent to separate analytical laboratories. The results of the split samples can be compared (e.g., calculate relative percent difference) to provide information on consistency and reproducibility of analytical procedures between the laboratories. However, due to the nature of air sampling canisters (different surface conditions, cleaning/certification procedures) and differences in analytical laboratory procedures (common in air analysis) the results are almost always meaningless. Please note that Air Toxics Ltd. does not recommend field splits and does not allow Air Toxics Ltd. canisters or other media to be sent to 3rd parties without obtaining prior written consent of Air Toxics Ltd.

#### 4.1.3 Field Blank

A field blank is a sample collected in the field from a certified air source. Analysis of the field blank can provide information on the decontamination procedures used in the field. Clean stainless steel or Teflon tubing and a certified regulator should be used. It is imperative that individually certified canisters (the sample canister and the source canister/cylinder, if applicable) be used to collect a field blank.

#### 4.1.4 Ambient Blank

An ambient blank is an ambient air grab sample collected in the field normally used in conjunction with soil gas or stationary source (e.g., SVE system) sampling. Analysis of the ambient blank can provide information on the ambient levels of site contaminants. It is imperative that an individually certified canister be used to collect an ambient blank.

#### 4.1.5 Trip Blank

When sampling for contaminants in water, the laboratory prepares a trip blank by filling a VOA vial with clean, de-ionized water. The trip blank is sent to the field in a cooler with new sample vials. After sampling, the filled sample vials are placed back in the cooler next to the trip blank and returned to the laboratory. Analysis of the trip blank provides information on decontamination and sample handling procedures in the field as well as the cleanliness of the cooler and packaging.

When sampling for compounds in air, a trip blank provides little, if any, of the information above. A trip blank canister can be individually certified, evacuated, and sent to the field in a box with the sample canisters. Since the valve is closed and the brass cap tightened, it is questionable if the trip blank canister contents are ever "exposed" to sampling conditions. At the laboratory, the trip blank canister will be pressurized prior to analysis with dry, zero air - a matrix that may be entirely different than the sampled air. The recovery of target compounds can vary by matrix (e.g., moisture, carbon dioxide) rendering the trip blank results meaningless. Air Toxics Ltd. does not recommend analyzing a trip blank for air sampling.

#### 4.1.6 Equipment Rinse

When sampling for contaminants in water, an equipment rinse is accomplished in the field by rinsing the decontaminated sampling equipment (e.g., bailer, submersible pump, tubing) with clean, de-jonized

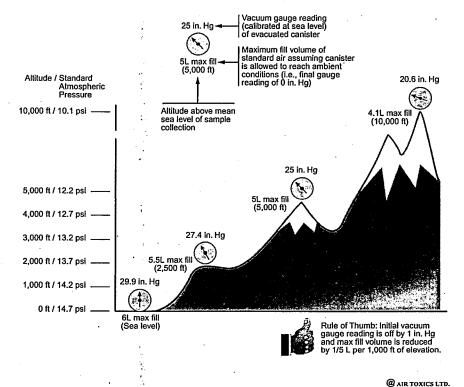
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water. A portion of the rinse water is collected in a VOA vial for analysis. The equipment rinse is similar to a field blank in that it provides information on decontamination procedures of sampling

When sampling for compounds in air, an equipment rinse can be used to determine if a sampling train has been properly decontaminated. Certified air is connected to the sampling train and fills an individually certified canister.

### 4.2 Considerations for Sampling at Altitude

Sampling at altitudes significantly above sea level is similar to sampling a stationary source under vacuum (see Section 4.3) in that target fill volumes may be difficult to achieve. The figure below illustrates the relationship between increasing altitude and decreasing atmospheric pressure. Ambient



conditions in Denver at 5,000 ft altitude are quite different than ambient conditions at sea level. Canister sampling is driven by the differential pressure between ambient conditions and the vacuum in the canister. There is less atmospheric pressure in Denver and 5 L is the maximum fill volume of standard air assuming the canister is allowed to reach ambient conditions (i.e., final gauge reading of 0 in. Hg). Theoretically, if you sample high enough (e.g., in space), no sample would enter the canister because there is no pressure difference between the evacuated canister and ambient conditions. To fill a canister to 6 L in Denver, you would need to use an air pump.

Sampling at altitude also affects gauge readings. The gauges supplied by Air Toxics Ltd. (see Section 2.2.4) measure canister vacuum relative to atmospheric pressure and are calibrated at approximately sea level. Before sampling at altitude, the gauges should be equilibrated (see Section 2.3.1). But even after equilibrating the gauge, verifying the initial vacuum of a canister at altitude is misleading. In Denver at 5,000 ft, expect the gauge to read 25, not 29.9 in. Hg. You do not have a bad canister (i.e., leaking or not evacuated properly). The canister is ready for sampling and the gauge is working properly.

Rule of Thumb: For every 1,000 ft of elevation, the gauge will be off by 1 in. Hg and the fill volume will be reduced by 1/5 L

If you have questions about sampling at altitude, please call Client Services at 800-985-5955.

### 4.3 Considerations for Soil/Landfill Gas Sampling

There are some additional sampling considerations for collecting grab samples (canister or Tedlar bag) from a soil boring, landfill boring, SVE system, or landfill gas (LFG) collection system. The general challenge with these samples arises from the need to employ long lengths of tubing to direct the soil gas, landfill gas, or process air to the canister or Tedlar bag. Tubing introduces the potential for contamination and diluting the sample. A good source of detailed information on soil gas sampling is contained in the ASTM D 5314 Standard Guide for Soil Gas Monitoring in the Vadose Zone.

- Use inert tübing. Teflon tubing is recommended. Tubing with an outer diameter of 1/4 in. works best with the fittings on the particulate filter.
- Do not reuse tubing. \$2 per foot for new tubing is a small price to pay for peace of mind.
- Purge tubing adequately. A long length of tubing has significant volume of "dead air" inside.
   Without purging, this air will enter the canister and dilute the sample. Consider using a hand-held PID/FID to confirm that you have purged the tubing and are drawing sample air through the tubing.
- Avoid leaks in the sampling train. Leaks of ambient air through fittings between pieces of the sampling train (e.g., tubing to particulate filter) will dilute the sample.

- Don't sample too fast. There is no established flow rate for collecting a soil gas or LFG sample, but sampling slower has advantages. First, any leaks in the sampling train will be less prominent at lower flow rates due to less differential pressure across the leaking connection. Second, sampling slower may allow the conditions in the vadose zone or landfill to equilibrate better and produce a more representative grab sample. Consider using a needle valve or even a 5 micron particulate filter (see Section 2.2.3) to reduce the flow rate into the canister or Tedlar bag.
- Purge the sample port. A sample port on a SVE system or LFG collection system can accumulate solids or liquids depending upon the location of the port in the process and the orientation of the port. An influent sample port located upstream of a filter or moisture knock-out can be laden with particulates or saturated with water vapor. Heavy particulate matter can clog the particulate filter and foul the canister valve. It is important to prevent liquids from entering the canister. The presence of water in a canister sample will significantly lower the recovery of both non-polar and polar compounds. A sample port oriented downward may have liquid standing in the valve. Purge the sample port adequately before connecting the sampling train.
- Consider the effects of sampling a process under vacuum or pressure. When collecting a grab sample from a stationary source such as an SVE system or LFG collection system, some sample ports may be under vacuum or pressure relative to ambient conditions. When the sample port is under vacuum, such as the header pipe from the extraction well network, it may be difficult to fill the canister with the desired volume of sample. A vacuum pump can be used to collect a canister grab sample from a sample port under considerable vacuum. See the related discussion on sampling at altitude in Section 4.2. When the sample port is under pressure, such as the effluent stack downstream of the blower and treatment system, you may inadvertently pressurize the canister. Only a DOT-approved sample cylinder should be used to transport pressurized air samples (see Section 4.4). Under no circumstances should an Air Toxics Ltd. canister be pressurized more than 5 psig for a 6 L canister and 15 psig for a 1 L canister. Bleed off excess pressure by opening the valve temporarily while monitoring the canister with a pressure gauge.

## 4.4 Considerations for Sample Cylinder Sampling

Sample cylinders, also known as "sample bombs", are DOT-approved, high pressure, thick-walled, stainless steel cylinders with a valve at each end. They were intended for collecting a pressurized sample for petroleum gas applications. Sample cylinders differ from sample canisters in that they do not have a Summa-passivated interior surface and are not evacuated prior to shipment. Sample cylinders are not suitable for analysis of hydrocarbons at ppbv levels. Sample cylinders can be used for analysis of natural gas by ASTM D-1945 and calculation of Btu by ASTM D-3588. Air Toxics Ltd.



assumes that clients requesting a sample cylinder have a pressurized process and sample port with a built-in gauge and 1/4 in. Swagelock fitting to attach to the sample cylinder. Air Toxics Ltd. has an inventory of 500 mL sample cylinders that are particularly suited for landfill gas collection systems (i.e., LFG to energy applications). This section provides step-by-step procedures for sampling with a sample cylinder.

#### Step-by-Step Procedures for Sample Cylinder Sampling

These procedures are for a typical stationary source sampling application and actual field conditions and procedures may vary. Follow all precautions in the site Health and Safety Plan when dealing with a pressurized sample port and sample cylinder.

- 1. Verify contents of the shipped package (e.g., chain-of-custody, sample cylinder, particulate filter)
- 2. Verify that gauge on sample port is working properly
- 3. Purge sample port
- 4. Remove brass caps on either end of cylinder
- 5. Attach particulate filter to upstream valve
- 6. Attach filter/cylinder assembly directly to the sample port
- 7. Open both valves 1/2 turn
- 8. Allow sample air to flow through sample cylinder (approximately 10 L for a 500 mL cylinder)
- 9. Close downstream valve of sample cylinder by hand tightening knob clockwise
- 10. Allow sample cylinder to pressurize to process pressure (max 100 psig)
- 11. Close upstream valve of sample cylinder and sample port
- 12. Detach filter/cylinder assembly from sample port and remove particulate filter
- 13. Replace brass caps
- 14. Fill out sample cylinder sample tag
- 15. Return sample cylinder in box provided
  - Unreturned sample cylinder charge of \$650 each.
- 16. Return sample media in packaging provided. Unreturned equipment charges:
  - \$45 per particulate filter
- 17. Fill out chain-of-custody and relinquish samples properly
- 18. Place chain-of-custody in box and retain pink copy
- 19. Tape box shut and affix custody seal (if applicable) across flap
- 20. Ship accordingly to meet method holding times

# Appendix B Geoprobe® Post-Run Tubing System

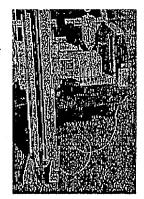


## **PRT Systems**

## Soil Gas Sampling Using the PRT System

The PRT system is the most common tooling system offered by Geoprobe Systems for soil gas "grab sampling". PRT stands for "post run tubing". It's a simple rool system for soil gas sampling that allows the user to drive probe rods to depth, then insert and connect inner tubing for soil gas sampling. Using an inner tubing for soil gas sampling. Using an inner tubing for soil gas sampling has many advantages; rod leakage is eliminated, dead volume that must be purged is reduced, and decon problems are reduced as the sample does not contact the rod bore. One of the biggest infrantages of this system is that the tubing is run in and connected for sampling after the rods have been driven to depth; there is no need to manage the tubing and maintain the connection during the driving process.

PRT adapters are comparible with a variety of tubing types to meet your soil gas sampling requirements.



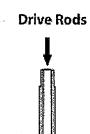
Post-flun Tubing (PRT) connected for soil gas sampling.

## **PRT Tubing** Adapters See page 7.6, Select the adapter for your tubing size. This adapter is inserted down the rod after it has been driven to depth. Standard **Probe Rods** 1 in., 1.25 in., or 1.5 in. See Chapter 2. **PRT Point Holders** and Points See page 7.5. A number of points and holders are available to sult your application.

The tools you'll need to perform soil gas sampling

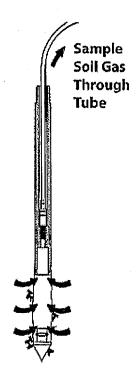
using "Post-Run" tubing

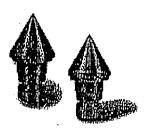
(PRT).





Insert



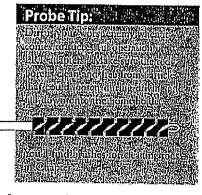


Expendable drive points for PRT soil gas sampling.

Rod emipped with a PRY point holder and expendable point are driven to depth.

Before the rods are pulled back, gas sampling tubing and a PRT adapter are inserted down the rod borg and connected to the point bodge. The rods are now pulled back (retracted) and soil gas is sampled through the tubing. Sample does not contact the rod bore,

Geoprobe Systems

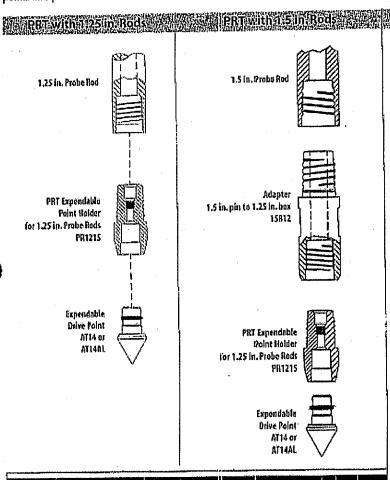


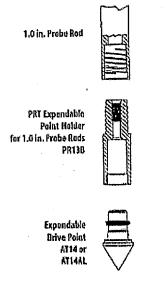
Geoprobe Systems"

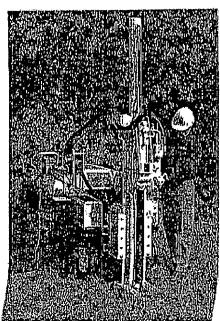
## **PRT Soil Gas Sampling Tool Configurations**

Our most popular configurations...

Geoprobe Systems offers both expendable and retractable point systems for PRT soil gas sampling. However, expendable point systems are by far the most commonly used. This is due to their simplicity, dependability, ease of use, and minimal decontamination requirements. Using the configurations shown below, the drive point is lost to the soil when the probe rod is retracted. In normal operations the probe rod is initially retracted a short distance, typically 6 in. (150 mm). In moist, fine grained soils that do not readily yield soil gas samples, the probe rods can be further retracted until a soil gas yielding zone is exposed. Note the use of O-rings on all points and point holders! This is a must for successful soil gas sampling.







Preparing to drive a 1.25 in, rod equipped with a PRT point holder and expendable drive point.

## PRT SolliGas Sampling Parks

**Point Holders** 

PRI 3B ......PRT Expendable Point Holder, threaded, for 1-in. Rod PRI 215 ......PRI' Expendable Point Holder, threaded, for 1.25-in. Rod PRIBE ................. O-rings for PRIBE Expendable Point Holder, Pkg. of 25

**Points** 

AT14 ...... Expendable Drive Points, Steel AT14AL ..... Expendable Drive Points, Aluminum

AT14R ............ O-rings for Expendable Drive Points, Pkg. of 25

Rod Adapter

15812 ..... Adapter, 1.5-in. pin to 1.25-in. box

AT1250R ........... O-rings for 1.25 in. Probe Rods, Pkg. of 25 ... O-rings for 1.5 in. Probe Rods, Pkg. of 25

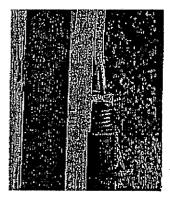
1-800-436-7762



## Selecting and Using a PRT Adapter

The PRT Adapter is a simple device that is sized to be press fit into a polymer tubing on one end, and then threaded (screwed) into the top of the point holder at the other end. "PRI" stands for "post-run tubing", this means that the tubing and adapter is inserted into the rod bore after the rods have been driven to their final sampling depth. You do not have to manage tubing as the rods are being driven.

All PRT adapters are left-hand threaded and must be rotated counter-clockwise to engage the point holder threads. PRT O-rings are soft, allowing the adapter to seal with minimal force.



PRT Adapter on soil gas sampling tubing ready to be inserted in rods.

RT Adapter Selection Guide	3 .			
PRT ADAPTER and PART NUMBER	RECOMMENDED ' TUBING SIZE	GEOPROBE® TUBING PART NO.	TUBING INTERNAL VOLUME (mL/II)	
Pn125	1/8 in. I.D. (3.2 mm I.D.)	TB12T Teffon*	2.41	
man thereof don't differentiam a feet	0.17 in. l.D. (4.3 mm l.D.)	TU174. LDPE	4,46	
PR175	3/16 fn. I.D. (4.8 mm I.D.)	T817T Teflon*	5.43	
PIRSS	1/4 in. l.D. (6.4 mm l.D.)	TB251, LDPE	9.65	
PR30S	5/16 In. I.D. (7.9 mm I.D.)	TB30T Teflon*	15.08	
PR25R O-Ring for all PRT Adapters (pack of 25)	Tubing At A Gl	lance		
OTE: The sorption characteristics of train tubings may not permit their use all soil gas sampling applications.	© (C	7L TB17T TB25	) ( L TB30T	TB37L

Tubing Sizes, Materials, and Specifics detailed on pg. 7.15.

## PRT Sampling Systems...

## More Parts, Accessories, and Tricks

## Retractable Drive Point Assembly AT21B

Stainless steel retractable point insert with 1:0 in, threaded point housing and ball bearings (for retaining the point insert). Point extends on pullback to allow gas sampling, but remains attached to holder for further probing. Point extends 2 inches (51 mm) on pullback. Can be easily disassembled for cleaning.

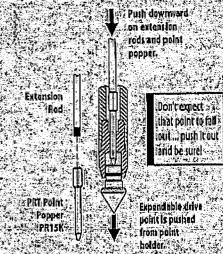


The AT21B retractable point is an alternative to expandable point systems.

Probe Rod

## Point Popper PR15K

Expendable or retractable points will not always freely disengage from the point holder at the rime of pullback. This is especially true when you have driven through moist clay zones and are using an O-ring to seal the rods. Under these conditions, a "point" popper" can be inserted on extension rods adown the probe rod bore. This assembly is used to up on the point and force it out of the point holder.





The Post-Run Point Popper (PR15X) dislodges Oving social points downhole.



AT211 ... Retractable Point Shaft cand AT218 ... O Rings

**Retractable Drive Point Parts** 

AT21B Retractable Drive Point Assembly

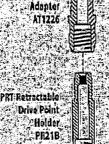
AT211B Retractable Point Housing
AT211 Stainless Retractable Point Shaft
AT212 Retractable Point Ball Bearing, Pkg. of 12,
AT21R O-Rings for Retractable Drive Point Pkg.

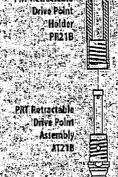
Point Assembly

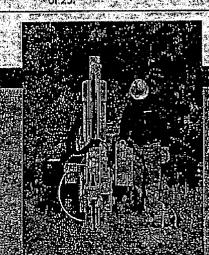
Sub-Assembly Parts



AT211B \_ Retractable Point Housing and AT212 ... Ball Dearings







## Probe Tip: Finding the Zone That Yields Soil Gas

Appendix C
Purge Volume Calculations



## Purge Volume Calculations

## Total Purge Volume = Tubing Volume + Pull Back Vold Volume + 20 mL

## 1. Tubing Volume for 1/8-Inch Inside Diameter Tubing

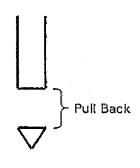
Volume =  $\pi \times r^2 \times h$ , where:  $\pi = 3.14$ , r = inside radius of tubing, and h = length of tubing. For r in inches and h in feet: Volume in mL (cubic centimeters) =  $3.14 \times r^2 \times h \times 196.64$ 

Length (feet)	Volume (mL)	Length (feet)	Volume (mL)	Length (feet)	Volume (mL)
1	2	19	46	37	89
2	5	20 .	48	38	92
3	7	21	51	39	94
4	10	22	53	40	96
5	12	23	55	41	99
6	14	24	58	42	101
7	17	25	60	43	104
8	19	26	63	44	106
9	22	27	65	45	108
10	24	28	67	46	111
11	27	29	70	47	113
12	29	30	72	48	116
13	31	31	75	49	118
14	34	32	77	50	121
15	36	33	80	51	123
16	39	34	82	52	125
17	41	35	84	53	128
18	43	36	87	54	130

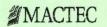
## 2. Vold space behind tip based on pull back

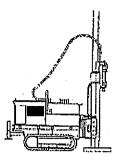
Volume =  $n \times r^2 \times h$ , where: n = 3.14, r = inside radius of tubing, and h = length of tubing. The table below was calculated based on rod/tip outside diameter of 3 cm. Tip shaft/rod inside diameter not included in calculation

Pull B	ack	Volume	Pull I	Back	Volume
Inches	cm	cc or mL	Inches	cm	cc or mL
0.5	1.27	9	5.5	13.97	99
1	2.54	18	6	15.24	108
1.5	3.81	27	6.5	16.51	117
2	5.08	36	7	17.78	126
2.5	6.35	45	7.5	19.05	135
3	7.62	54	8	20,32	144
3.5	8.89	63	8.5	21.59	153
4	10.16	72	9	22.86	162
4.5	11.43	81	9.5	24.13	170
5	12.7	90	10	25.4	179



Appendix D
Vacuum Testing Procedure



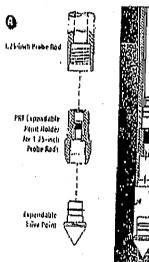


## Green Services, Inc.

## Geoprobem Soil Gas Sampling

Prior to the introduction of Post Run Tubing (PRT) soil gas sampling there were less effective ways to collect soil gas samples with a Geoprobe. An expendable point was driven to the sampling depth and detached by withdrawing the drive rods far enough to create an opening for gas to move into the drive rods from the base of the hole (this has not changed). Then soil gas samples were withdrawn by attaching tubing to the top of the drive rods and applying a vacuum. The large inside diameter (ID) of the drive rods required a large purge volume, and produced samples that were not uniformly representative of contaminant concentrations in the subsurface. Small ID tubing was needed to reduce the purge volume, and was incorporated into the sampling system with an inflatable packer to provide the seal at depth and insure the collection of representative samples'. The packer system functioned well, however it was cumbersome in the field, and had problems with carryover contamination and possible leaks in the pressurized system.

When Geoprobe introduced the PRT system there was doubt that the seal created with



the O-ring was reliable. A test was designed and repeated to determine the reliability of the seal. A drive rod was attached to the point holder, and a PRT adapter with tubing was assembled and installed, as if to collect a sample (as shown). The annular space between the tubing and drive rod ID was then filled with water. A vacuum was then applied to the tubing (as if collecting a sample), and even with the open end of the point holder blocked (where soil gas would enter the system to fill the vacuum), no water was forced through the O-ring seal.

On site this test can be repeated without the water. The assembled system will be sealed (open end of the point holder blocked) and a vacuum applied with a syringe. When the syringe is released, any gases leaked will displace the syringe plunger from its original position. Close

attention to detail will be needed when performing this test to prevent leakage of air into the system from the blocked end of the point holder, and not to exceed reasonable vacuum levels that are used to collect samples.

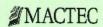
Powerful Tools

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Procedure Prom Target Environmental Services.

Appendix E NIOSH Method 1550



## **NAPHTHAS**

Table 1

MW: Table 1

CAS: Table 1

RTECS: Table 1

METHOD: 1550, Issue 2

**EVALUATION: FULL** 

Issue 1: 15 February 1984

Issue 2: 15 August 1994

OSHA: Table 1 NIOSH: Table 1 ACGIH: Table 1 PROPERTIES: Table 1

**SYNONYMS:** Petroleum ether (benzin), rubber solvent, petroleum naphtha, VM&P naphtha, mineral spirits, Stoddard solvent, kerosene (kerosine), coal tar naphtha.

	SAMPLING	MEASUREMENT			
SAMPLER:	SOLID SORBENT TUBE (coconut shell charcoal, 100 mg/50 mg)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID		
•	(caccong change and,	ANALYTE:	naphtha hydrocarbons		
FLOW RATE:	0.01 to 0.2 L/min	DECORPTION			
VOL-MIN:	1.3 L @ 400 mg/m <sup>3</sup> ; 0.2 L @ 2500 mg/m <sup>3</sup>	DESORPTION:	1 mL CS <sub>2</sub> ; stand 30 min		
-MAX:	20 L @ 400 mg/m³; 3.2 L @ 2500 mg/m³	INJECTION VOLUME: 5 μL (packed column); 0.1 to 1 (capillary column)			
SHIPMENT:	routine	1	, , , ,		
SAMPLE		TEMPERATURE-I	INJECTION: 200 to 250 °C DETECTOR: 250 to 300 °C		
STABILITY:	at least 1 week @ 25 °C		-COLUMN: 50 to 250 °C @ 8 °/min		
BLANKS:	2 to 10 field blanks per set	CARRIER GAS:	N <sub>2</sub> or He, 30 mL/min		
BULK SAMPLE:	C SAMPLE: required; 5 to 10 mL		glass, 3 m x 6-mm, 10% SP-2100 on Supelcoport 80/100 or 30-m fused silica		
	ACCURACY		capillary, 0.325-mm ID, 1.0-µm DB-1 or equivalent		
RANGE STUDIE	D: see EVALUATION OF METHOD	CALIBRATION:	solutions of bulk naphtha in CS <sub>2</sub>		
BIAS:	see EVALUATION OF METHOD	RANGE:	0.5 to 10 mg per sample [2,3,4]		
OVERALL PRECISION (Ŝ <sub>rT</sub> ): 0.05 [1]		ESTIMATED LOD	0: 0.1 mg per sample		
ACCURACY:	see EVALUATION OF METHOD	PRECISION (S <sub>r</sub> ):	0.01 [1]		

**APPLICABILITY:** The working range is 100 to 2000 mg/m<sup>3</sup> for a 5-L air sample. This is a general procedure for analysis of various types of hydrocarbon mixtures called "naphthas" which are used as thinners in paints and varnishes and as general purpose solvents.

INTERFERENCES: Most naphthas are quite complex. The components elute over a wide temperature range by gas chromatography, making interferences from other substances possible. Columns and conditions must be chosen to obtain the desired degree of separation for a given mixture.

OTHER METHODS: This method combines and replaces Methods S86 [2], S380 [3] and S382 [4]. A similar method appears in the criteria document [5].

#### REAGENTS:

 Eluent: Carbon disulfide\*, chromatographic quality, containing 0.1% (v/v) octane, 0.5% (v/v) hexadecane or other suitable internal standard.

NOTE 1: Use an internal standard which is

not a major constituent of the sample.

NOTE 2: Use toluene in place of carbon

disulfide for low-boiling analytes [5].

2. Naphtha bulk sample.

3. Nitrogen or helium, purified.

4. Hydrogen, prepurified.

5. Air, filtered.

\* See SPECIAL PRECAUTIONS.

#### **EQUIPMENT:**

- Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, containing two sections of activated (600 °C) coconut shell charcoal (front = 100 mg; back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
- 2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
- 3. Gas chromatograph, FID, integrator and column (page 1550-1).
- 4. Vials, glass, 2-mL, PTFE-lined crimp caps.
- 5. Syringe, 10-μL (1-μL syringe for capillary columns) and other convenient sizes for preparing standards, readable to 0.1 μL.
- 6. Volumetric flasks, 10-mL.
- 7. Pipet, delivery, 1.0-mL, with pipet bulb.

**SPECIAL PRECAUTIONS:** Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C); work with it only in a hood.

#### SAMPLING:

- 1. Calibrate each personal sampling pump with a representative sampler in line.
- 2. Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
- 3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size which contains between 0.5 and 8 mg naphtha.
- 4. Cap the samplers with plastic (not rubber) caps and pack securely for shipment. Ship a bulk sample (5 to 10 mL) in a separate container from the sorbent tubes.

#### **SAMPLE PREPARATION:**

- Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
- 6. Add 1.0 mL eluent to each vial. Attach crimp cap to each vial.
- 7. Allow to stand 30 min with occasional agitation.

#### **CALIBRATION AND QUALITY CONTROL:**

- 8. Calibrate daily with at least six working standards over the range 0.1 to 10 mg naphtha per sample.
  - Add known amounts of naphtha bulk sample to eluent in 10-mL volumetric flasks and dilute to the mark.

- b. Analyze together with samples and blanks (steps 11 and 12).
- c. Prepare calibration graph (ratio of peak area of analyte to peak area of internal standard vs. mg naphtha).
- Determine desorption efficiency (DE) at least once for each batch of charcoal used for sampling in the calibration range (step 8). Prepare three tubes at each of five levels plus three media blanks.
  - a. Remove and discard back sorbent section of a media blank sampler.
  - Inject a known amount of naphtha bulk sample directly onto front sorbent section with a microliter syringe.
  - c. Cap the tube. Allow to stand overnight.
  - d. Desorb (steps 5 through 7) and analyze together with working standards (steps 11 and 12).
  - e. Prepare a graph of DE vs. mg naphtha recovered.
- 10. Analyze three quality control blind spikes and three analyst spikes to insure that the calibration graph and DE graph are in control.

#### **MEASUREMENT:**

- 11. Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1550-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
  - NOTE 1: The columns and conditions given provide moderate to good separation of components. If less resolution is needed, use shorter, less efficient columns as were used in validation of Methods S86 [2], S380 [3] and S382 [4].
    - NOTE 2: If peak area is above the linear range of the working standards, dilute with eluent, reanalyze and apply the appropriate dilution factor in calculations.
- 12. Measure peak area. Divide the peak area of analyte by the peak area of internal standard on the same chromatogram.

#### **CALCULATIONS:**

- 13. Determine the mass, mg (corrected for DE) of naphtha found in the sample front (W <sub>f</sub>) and back (W<sub>b</sub>) sorbent sections, and in the average media blank front (B <sub>f</sub>) and back (B<sub>b</sub>) sorbent sections. NOTE: If W<sub>b</sub> > W<sub>b</sub>/10, report breakthrough and possible sample loss.
- 14. Calculate concentration, C, of naphtha in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V}, mg/m^3.$$

#### **EVALUATION OF METHOD:**

Methods S86 (Naphtha, Coal Tar), S380 (Petroleum distillate) and S382 (Stoddard Solvent) were issued on March 14, 1975 [2,3,4]. They were validated at 24 °C and approximately 755 mm Hg using 10-, 4- and 3-L air samples, respectively, of 2-50-W Hi-Flash Solvent (Neville Chemical Co.; BP 154 to 195 °C; d 0.893 g/mL), VM&P Naphtha (Amsco Product 1101; BP 120 to 147 °C; d 0.743 g/mL) and Stoddard Solvent (Fisher Scientific Co.; BP 159 to 176 °C; d 0.774 g/mL) [1]. Overall precision and recovery were as shown below, representing a non-significant bias in each method. Breakthrough tests in dry air showed a capacity of 20 to 25 mg of each solvent tested. Capacity at high relative humidity was not determined.

	Overall		Range	Studied		Avg.	•
Method	Precision (Ŝ <sub>rī</sub> )	Bias	Accuracy	mg/m³	mg per sample	DĔ	Ref.
S86 <sup>(b)</sup> S380 <sup>(a)</sup>	0.051 0.052	5.99% -4.37%	±15.0% ±12.5%	193 to 809 937 to 3930	2 to 8 4 to 16	0.88 0.96	[1,2] [1,3]
S382 <sup>(b)</sup>	0.052	-3.10%	. ±11.4%	1417 to 5940		0.95	[1,4]

### NOTES:

- (a) Data based on experiments using an internal standard method with a 10 ft., 1/8" stainless steel column packed with 10% OV-101 on 100/120 mesh Supelcoport.
- (b) Data based on experiments using an internal standard method with a 6 ft., 1/8" stainless steel column packed with 1.5% OV-101 on 100/120 mesh Chromosorb W.

#### REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S86, S380, S382, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 2, Method S86, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [3] Ibid, V. 3, S380, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [4] Ibid, S382.
- [5] Criteria for a Recommended Standard...Occupational Exposure to Refined Petroleum Solvents, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-192 (July, 1977).

#### **METHOD REVISED BY:**

Ardith A. Grote, NIOSH/DPSE; S86, S380 and S382 originally validated under NIOSH Contract CDC-99-74-55.

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Limits, mg/m³ NAME OSHA		Vapor	<del>-</del>
CAS # Predominant	Boiling NIOSH	Pressure, kPa	Liquid Density
RETECS Hydrocarbon Species	Range (°C) ACGIH	(mm Hg) @ 20 °C	(g/mL @ 15 °C)
Petroleum ether (a)	30 to 60	13 (100)	0.63 to 0.66
8032-32-4 Ol6180000	C <sub>5</sub> -C <sub>6</sub>	(13 °C)	aliphatic 350; C 1800
0.0100000			
Rubber solvent (a)	45 to 125 C <sub>5</sub> -C <sub>8</sub>	(c) 2000 (500 ppm)	0.67 to 0.85
SE7449000 8030-30-6	O <sub>5</sub> -O <sub>8</sub>	2000 (300 ppm)	aliphatic 350; C 1800
			1590 (400 ppm)
Petroleum naphtha (b)	30 to 238 C <sub>8</sub> -C <sub>8</sub>	5 (40) 2000 (500 ppm)	0.6 to 0.8
(Petroleum distillates mixture)	O <sub>6</sub> -O <sub>8</sub>	2000 (000 μμπ)	250, 0 4000
8002-05-9 SE7449000			350; C 1800 
VM&P naphtha (a)	95 to 160 C <sub>7</sub> -C <sub>11</sub>	0.3 to 3 (2 to 20)	0.72 to 0.76
8032-32-4 Ol6180000			<20% aromatic350, C 1800 1370 (300 ppm)
Mineral spirits (a)	150 to 200 C <sub>9</sub> -C <sub>12</sub>	0.2758(2) 2900 (500 ppm)	0.77 to 0.81
8052-41-3	Og*O <sub>12</sub>	2900 (000 ppiii)	<20% aromatic
WJ89250000			350; C 1800 525 (100 ppm)
Stoddard solvent (a)	150 to 210	0.2758 (2)	0.75 to 0.80
8052-41-3	C <sub>9</sub> -C <sub>12</sub>	2900 (500 ppm)	<20% aromatic
WJ8925000			350; C 1800 525 (100 ppm)
Kerosene (a)	175 to 325	(c)	0.8
8008-20-6	C <sub>9</sub> -C <sub>16</sub>	-	<20% aromatic
OA5500000			100
Coal tar naphtha (b)	110 to 190 C <sub>8</sub> -C <sub>10</sub>	<0.7 (<5) 400 (100 ppm)	0.86 to 0.89

## NAPHTHAS: METHOD 1550, Issue 2, dated 15 August 1994 - Page 5 of 5

8030-30-6

aromatic 400 (100 ppm)

DE3030000 (NIOSH)

- (a) As defined by NIOSH Criteria Document [5].
- (b) As defined for OSHA PEL.(c) Not available.

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